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Telephone: FLEet Street 3212 (26 lines)

Telegrams: Allangas - Fleet - London

Editor  
M. C. HYDE

Manager  
H. A. WILLMOTT

Director N. B. LIVINGSTONE WALLACE

**Midland Office**

Daimler House, Paradise Street,  
Birmingham. [Midland 0784-5]

**Leeds Office**

Permanent House, The Headrow,  
Leeds 1. [Leeds 22601]

**Scottish Office**

116 Hope Street, Glasgow C2.  
[Central 3954-5]

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# CHEMICAL AGE

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## ALICE IN POLYMERLAND

**A**STOUNDED'. 'A bitter blow'. 'If these polymers aren't "synthetic organic chemicals" then what on earth are they'? These were some of the immediate reactions of chemical industry leaders at the decision of the arbitration tribunal that four polymers are not 'synthetic organic chemicals' within the meaning of the 1921 Safeguarding of Industries Act and therefore had been properly excluded by the Board of Trade from Key Industry Duty List J.

Sir Miles Thomas (Monsanto Chemicals) and Mr. Peter Delafield (Distillers Plastics Group) had every reason to be astounded, for this was an astonishing decision that will have far-reaching consequences. We have been able to find few chemists, working on polymers or not, who support Sir Christopher Ingold's view that polythene, polystyrene, p.v.c. and polymethyl methacrylate, are not 'synthetic organic chemicals'. How is it then that a tribunal can decide that they are not 'synthetic organic chemicals'?

It is one of the absurdities of the situation that the Board of Trade has always interpreted the term under the 1921 Act as including polymers, for melamine formaldehyde, oxamide, cellulose acetate, etc., have been on the K.I.D. list for more than 30 years. At no time since 1921, the period during which the great U.K. polymer industry has been built up, has the opportunity been taken to clarify the position. The few thermoplastic polymers that were in production then were classified as 'synthetic organic chemicals'; polymers that have been developed since have been kept out.

The U.K. Tariff is to be superseded in January by the Brussels Nomenclature and the polymers concerned, with plastics materials, are to be transferred from their present classification of 'n.e.s.' to a new section 'plastics', but still at the rate of 10%.

In the U.S. tariff these polymers are rightly classified as 'synthetic organic chemicals'. Exports of them leave that country and cross the Atlantic as 'synthetic organic chemicals', but on arrival in this country they are no longer chemicals. At the tribunal, much was made of the fact that the big companies have split their operations into chemical and plastics divisions or groups and that this showed that they looked on polymers as something different from chemicals. This argument is fallacious. No one would suggest that dyestuffs are not chemicals because I.C.I. have a Dyestuffs Division or that fertilisers are not chemicals because they have a fertiliser group. This was one of the many red-herrings that came up at the hearing.

The decision, however, has been made and there is no appeal against it. We cannot help feeling that it does not matter much how these materials are classified, provided that a vital section of the U.K. industry is not allowed to become vitiated because it is shut out of the world's major polymer markets at the same time that its home market is open to mounting competition.

It was said, somewhat sarcastically, that the big four companies, because they were seeking to have polymers defined as 'synthetic organic chemicals' and, therefore, subject to the higher duty of 33½%, were 'tender plants' in need of protection. We see no justification for that point of view, for

each of the companies has proved well able to withstand competition; in fact in the highly competitive chemical field these companies have pioneered major developments in the polymer field.

This was another red herring which does not hide the fact that so far as p.v.c. is concerned, the U.K. industry has been subjected to rising low-priced imports. The following are current estimates of U.K. production and imports of polymers:

	U.K. Output Tons	Imports Tons
P.V.C. ....	68,000	12,000
Polythene ....	65,000	4,500
Polystyrene ....	29,000	715
Polymethyl-methacrylate ....	15,000	1,500

It was argued that total imports of the four polymers did not amount to much when compared with U.K. production of them as a whole. Imports of p.v.c., which will doubtless rise faster following the decision, belie this view. The imported price, quoted at the tribunal, was 1s. 4d. per lb. after payment of freight and the 10% tariff, against a U.K. delivered price of 1s. 6½d.

Of most concern have been imports of what has been described as 'subsidised' p.v.c. from Japan and Italy. Japanese capacity totals 200,000 tons a year, although at present only about half of it is being utilised. The Japanese home market is taking something like 40,000 tons a year, leaving an actual export surplus of 60,000 tons, or nearly the equivalent of the total U.K. output. This massive capacity has been made possible only because of the need of Japan's rayon industry for caustic soda; the ensuing build-up of extremely large chlorine capacity has been utilised for the production of p.v.c. Hydro-electric power is plentiful in Japan and low-labour costs combine to give that country's p.v.c. a distinct price advantage, even when shipped to this country. Already, moves have been made in Australia to protect that country's young and growing p.v.c. industry from the Japanese product.

Italy, the other major exporter of p.v.c., bases its production on the State-owned natural gas reserves. There is also the unusual arrangement whereby vinyl chloride made by the State A.N.I.C. is shipped to Wacker in West Germany where it is polymerised.

Although they have to contend with these low-priced polymers competing in their own home market, British producers are virtually barred from exporting to the major markets because none of them has such low tariffs as the U.K. On polymers, the U.S. tariff is 25-35%; France, 30%; Italy, 25%; Japan, 20%; Germany, 11-14%.

While it would be entirely wrong for the British chemical industry to become protectionist, it is extremely unjust that it should be faced with this type of competition.

At present, p.v.c. is the main bone of contention, but within two years the U.K. polythene producers will also be affected. Already the U.S. have a capacity of 150,000 tons a year and an export surplus in excess of U.K. productive capacity. U.S. producers also have the advantage of complete protection from British polythene. All over the world, polythene facilities are being expanded and once national demands have been met, a vast exportable surplus will become available and the most profitable world market open to it will be Britain.

At the same time, foreign-based firms have with active B.o.T. encouragement set up polythene plants in the U.K. It is no wonder that U.S. companies no longer look on the U.K. as a good investment for a European production base. Understandably they are turning their attention to the Common-market area. Polymer factories built there would give the firms concerned a home market roughly the size of the U.S., with the U.K. as a large export market. The eventual 22% tariff of the Common-market will protect them effectively from any possible U.K. competition.

The U.K. polymer industry would then be excluded from the main European markets as well as from the U.S. and Japan. This kind of economic policy is suicidal. Quite apart from the serious immediate effect on U.K. polymer producers, is the possible impact on this country's whole petrochemical industry.

The smallest economic production for a cracker is 30,000 tons a year. Outputs of that order must be backed by a stable home market in the main products made from ethylene and propylene. There is now no such assurance of a firm home market for polythene, styrene or methyl methacrylate. There will be little incentive to expand capacities of these chemicals or to invest large sums in new polymers—for which there is an undoubted future—such as polycarbonates or Delrin. It would be most unfortunate if this country were to fall behind others in the development of new polymers because the Government refused to provide the producers with a stable home market.

There is a pressing need for action to prevent the run-down, or even partial run-down, of a vital British industry. The way to do this is to give U.K. polymer producers parity of competition; in view of the gravity of the present situation the Government should give immediate attention to the matter.

## LITHIUM DISPERSIONS

**D**ISPERSIONS of lithium metal, lithium hydride and solutions of lithium alkyls which are extremely pure, have been developed by Foote Mineral, Philadelphia, US. The compounds are claimed to contain less than 0.1 per cent of oxygen-containing compounds, and are suggested as being particularly useful for catalytic work. Because of the low content of oxygen-containing compounds which coat the lithium particle, the induction period to reaction has been eliminated. Reaction begins immediately and continues to completion, so reducing the risk of explosion and fire. Also, in the preparation of the new compounds, dispersal aids such as oleic and stearic acid are unwanted.

Reactiveness of the lithium dispersions is indicated by their spontaneous ignition in water. Their reactivity is, in fact, a problem now taxing Foote Mineral's chemists, with particular regard to the most suitable container for transport of these pure compounds. Any container used for the dispersions which is not free of contaminants, or if it reacts with the lithium, causes the lithium reactivity to be decreased. The containers must, of course, be dry.

In the technique used to prepare lithium dispersions,

reported by Foote Mineral's research chemist, W. Borkowski, hydrocarbons (unspecified) are used as vehicles for the lithium. The hydrocarbon is subjected to purification to remove oxygen- or nitrogen-containing compounds. The dispersions are then prepared by using high speed agitation and temperatures of about 200°C. Particle size of the dispersions ranges from 15 to 50 microns. Hydride dispersions are produced directly from lithium metal dispersion, and lithium alkyls by direct reaction with alkyl halides. In this manner, the lithium remains out of contact with moisture or air.

The new lithium dispersions are now being evaluated by various US companies. One likely application is their use in Ziegler, Grignard, Wurtz, Friedel-Crafts and other organic reactions. Another use stressed by Borkowski is in catalytic polymerisations, as for instance, in isoprene polymerisations.

Dispersed lithium hydride, it is believed, may supplant not only other alkali metal hydrides, but also the complex metal hydrides, such as lithium aluminium hydride and lithium borohydride.

# 'If Polymers Are Not Synthetic Organic Chemicals What On Earth Are They?'

'Dumbfounded', says Mr. Delafield: 'A Bitter Blow', says Sir Miles

**N**EWs that the decision of the polymer arbitration tribunal had gone against the complainants—Bakelite, Distillers Co., I.C.I. and Monsanto Chemicals—was announced briefly in last week's 'Chemical Age'. The ruling, which means that polythene, polystyrene, p.v.c. and polymethyl methacrylate are not 'synthetic organic chemicals' under the Safeguarding of Industries Act 1921 and are therefore not eligible for a 33½% duty under Key Industry Duty List J, is final and there is no appeal against it.

Shocked by the decision which they were sure would go in their favour and alarmed at the grave prospect of a steep rise in imports of cheap polymers while their products are virtually excluded from the major polymer producing countries, representatives of the four companies gave their reactions to 'Chemical Age'. One of the first comments came from Mr. Peter Delafield, managing director of British Resin Products Ltd., who said Distillers were completely dumbfounded at the ruling. 'If,' he added, 'these four polymers are not "synthetic organic chemicals", then what on earth are they?'

Sir Miles Thomas told us that Monsanto Chemicals were 'astounded at the decision, particularly as other and similar polymers are already subject to the Key Industry Duty. If the four polymers which formed the subject of the enquiry are not "synthetic organic chemicals", what in heaven's name are they?'

He pointed out that Monsanto were currently making a very large investment in polymer manufacture in this country, as were several other leading British chemical manufacturers. Sir Miles added: 'The decision of the tribunal is a bitter blow.'

I.C.I., in a statement to CHEMICAL AGE, wanted to clear up a misunderstanding about the decision. Although its effect was that the polymers would not be given K.I.D. protection, the tribunal was only concerned with the question of definition and could not touch the broad issue of whether, in principle, the polymers warranted increased protection.

## Tariff Handicap

A company spokesman told us: 'I.C.I. consider themselves as efficient as any of the world's polymer producers and, in fact, were a leader in discovery and development in this field. However, they share with other U.K. manufacturers of polymers the handicap that their overseas competitors enjoy higher tariff protection in their home markets than is afforded to U.K. manufacturers.'

'The present duty of 10% on the imports of these polymers into the U.K. compares with an import tariff of 25% to 35% in the U.S., 22% to 27% in Italy, 30% in France, 20% in Japan, and 11% to 14% in Germany. The effect is that overseas competitors can ship materials into the U.K. market while a U.K. producer cannot profitably export into these high tariff countries. If it were possible to sell in the home markets of our overseas competitors on the same terms that they can sell here, there would be no need for U.K. plastics producers to seek higher rates of duty.'

When approached for their view, Bakelite Ltd. said they fully supported the I.C.I. statement.

The tribunal, comprising Mr. A. W. Roskill, Q.C., and two London University professors of chemistry, Professor E. D. Hughes and Professor E. E. Turner, heard evidence on seven days. It had been agreed that the costs of the hearing, and of a preliminary hearing, would be borne in equal shares by the complainants, the Board of Trade as

respondents and the companies who were the opponents in the case.

Extensive reports of the tribunal's proceedings have appeared exclusively in 'Chemical Age' of 15, 22 and 29 November. Our final report appears in this issue. Apart from the official transcript of the proceedings, there is no other record of the hearing.

The complainants were represented by Sir Lionel Heald, Q.C., with Mr. S. Gratwick; the Board of Trade by Mr. K. Johnston, Q.C., with Mr. R. Winn; and the opponents by Mr. J. P. Graham, Q.C., with Mr. G. D. Everington. The opponents were: Anglo-American Plastics Ltd., British Cellophane Ltd., Chiltern Hunt Ltd., Henry Jackson (Liverpool) Ltd., Metal Box Co. Ltd., Armoide Ltd., Commercial Plastics Ltd., Duraplex (Plastics) Ltd., Rubber Improvements Ltd., Stanley Smith and Co. Ltd., Storeys Ltd., and Wallington Weston and Co. Ltd.

## C.A. Feature was 'Piece of Lobbying', Board of Trade Q.C.

**M**AKING his final submission for the Board of Trade, Mr. Johnston said that the first matter he would like to deal with arose from the suggestion that there should be or might be some witness from the B.o.T. Mr. Johnston submitted that it was very difficult to see how a B.o.T. official could really help the tribunal on the matter that had to be decided.

The B.o.T. might have their views but they were of no value to the tribunal and could not be given in evidence as far as the law was concerned as regards the construction of the section; as far as the other side was concerned, technical advice had been called.

What had happened was that some questions had been put to Sir Christopher Ingold—he supposed as an ordinary citizen rather than as an expert—namely, what he thought of the terrible position that all those things should be on the list and that the Customs had been 'illegally exacting tribute' on those goods like cellulose acetate and so forth. 'It is now said that it should not have been on the list at all, that it was illegal. Of course, that point does not really bear examination. The moment the list is made, it is the law; because the Act says so.'

Referee: 'Subject to anything Sir Lionel may have to say, I felt that the adverb at the time was not perhaps wholly accurate.'

Literally, it could not be true, said Mr. Johnston. No doubt what would be said was that it could be a justification, as it now appeared, or without justification, if the B.o.T. were right in their present argument or something of that kind.

In his submission, when the tribunal looked at this legislation it was perfectly plain and it was recognised that the B.o.T. in compiling their list might take the view which would be open to correction, because it was provided in the Act that within three months people could object to certain things being on the list. That recognised that the B.o.T.'s view was not final. It might be adjudicated upon and reversed. From past decisions, in fact, materials and things had been taken off the original list and Mr. Johnston instanced pinene where the B.o.T. had given in.

So far as artificial camphor had been concerned, it was not synthetic. The question of whether it was a chemical had not been tested. If the B.o.T. came to a certain view it might be corrected. 'If, after three months, the position goes solid, it is then on the list for good or ill. And, as I then pointed out, until 1936 it could not be got off again.'

What he had overlooked in remarking on vested interests (see CHEMICAL AGE, 22 November, p. 857) and which arose out of an observation made by the referee, was that by 1936, there had



been passed the Import Duties Act, 1932. That Act was really rather a landmark in U.K. economic history. The first section referred to a 10% *ad valorem* customs duty which was imposed on everything.

If the B.O.T. at any time after 1936 took something off the list because on consideration it appeared to them that it was not properly on, then immediate questions arose under Section 3(5) of the Import Duties Act on the duty ceasing to be chargeable; that was to say it was open to the Committee as it was then to put on any duty they liked on the ground, or because it appeared to the committee to be advisable, that it was in the interests of trade and industry of the U.K.

Referee: 'A complete answer to that comment, because it is no more than comment, would be what you have already said, if it is right, is it not, namely that if no objection is made within the time limited by the Safeguarding of Industries Act the composition hardens, and although something might have got on the list wrongly it is there.'

Mr. Johnston: 'It is there.'

Referee: 'So the Board of Trade hands are tied in that respect, is not that right?'

### Ample Powers to Restore Duty

Mr. Johnston said it was certainly right in 1936. No doubt having had power, once they had power to amend, it would be within their powers to strike things off of their own volition. He was only pointing out that if they did that, not only did they hit at a vested interest but the vested interest would be expecting to come and say to the committee that it was in the interests of trade that they should go on being protected. Therefore there were ample powers at any rate to restore the duty in whole or in part.

Anything of that sort had nothing to do with the present question and that what the list had to do was to list articles falling under the general description and it was a list which was either proper or improper according to whether the things were rightly included or rightly excluded. 'That is the sole question, and it is for that reason that we say and submit that the fact that 25 years ago in some cases, and 35 years ago in other cases, the Board of Trade put on things which according to their argument now they are saying ought not to have been on, it is with respect neither here nor there. The Board of Trade may have made a mistake. The Act recognises the possibility that any view they take may be open to correction.'

On the question of the changed view regarding the formula of oxamide, Mr. Johnston said it might be that things had changed, but it had to be remembered that over a large period of years the views about substances and what they were might very well change.

Referee: 'I suppose the character of the substance can change, and a fine

chemical might cease to be a fine chemical.'

Therefore, said Mr. Johnston, he submitted that the B.O.T. views from time to time, which were all those things represented, were not germane to any issue that arose before the referee at all.

Mr. Graham had asked Mr. Johnston to deal with two matters that Mr. Graham had by-passed. One was the word 'chemicals,' which when it came to fine chemicals also included the elements. It was true that synthetic organic chemical could not include elements, but the word 'chemicals' might, and that appeared in fine chemicals where it appeared in the other part. That was why one got the word 'chemicals' and not chemical compounds.

The other matter was concerned with 'The Chemical Age' which was A.11 of the exhibits. It was a 'piece of lobbying.'

Sir Lionel: 'A piece of history, not lobbying.'

Referee: 'Lobbying and history.'

Sir Lionel: 'Very much so.'

Mr. Johnston referred to the observations attributed to Dr. Henry Dreyfus in CHEMICAL AGE, 19 February 1921. Dr. Dreyfus was, he said, referring to the shortage of acetone and acetic acid (solvents for cellulose acetate). It might be, said Mr. Johnston, that Dreyfus was regarding the cellulose acetate manufacture itself as a key industry, but it was

not going to be described as a chemical industry. The inference was perfectly plain. What Dr. Dreyfus was talking about were those particular chemicals which were plainly in the context, the acetone, acetic acid and methyl acetate. Dr. Dreyfus had used the word in the proper way and that was the industry that should be encouraged.

Mr. Johnston said, in his submission, what Dr. Dreyfus was worrying about were the chemicals, i.e., the acetone, and acetic acid and so forth.

Another point on this article in CHEMICAL AGE concerned the gas mantle industry.

In conclusion, it is submitted that the manufacturers of thorium and rare-earth salts, incandescent mantles, and clay rings be recognised and assisted as "Key" industries.'

What Parliament had done was to take the chemicals, they had taken thorium and rare-earth salts, but not the manufactured articles.

The persons putting forward the views in the CHEMICAL AGE article, were concerned, maintained Mr. Johnston, with the chemicals. In the case of gas mantles it was the materials thorium and rare-earth salts and they were not concerned with things made from those materials or anything like that. 'We say, therefore, that on balance this particular article in so far as it helps you at all is in our favour, and does indicate, as we submit, what "chemical" means in the Act.'

This ended Mr. Johnston's final submission.

## Real Controversy was Over Use of 'Chemicals', says Sir Lionel Heald

ONE very important point which Sir Lionel Heald said he wished to draw attention to in his final submission on behalf of the complainants was that the argument had been conducted on the basis of chemical evidence and chemical definition by experts in organic chemistry. These experts had all agreed, Sir Lionel said, that the type of language used in this schedule was not the type of language to which they were accustomed. He asked how anybody could say today what exactly was the ambit of the synthetic organic chemical industry in 1921.

The first thing that had to be done in his submission in the present case was to look at the expression 'synthetic organic chemical', and at the word 'chemicals'. Why had Parliament not used the expression 'chemical compounds', which was what Sir Christopher Ingold would have advised them to do if he had been present in 1921.

One of the questions the tribunal had to ask was why they used that word and he, Sir Lionel, could provide one very good answer; for the very reason that Parliament did not want to have meticulous argument such as the tribunal had had to deal with. They wanted this to be a statute which could be administered by the Customs in a practical business-like way.

Referee: 'They might have been in some difficulty because of the distinction

with regard to elements'. That was what Mr. Johnston had drawn attention to in his submission.

Sir Lionel said that 'Chemicals are simply elements or combinations of elements'. The hearing was quite clear that polymers were combinations of elements. That was why Parliament used the word 'chemicals' just in order to avoid that kind of hair-splitting. Nobody in the present case had seriously suggested also that the word 'organic' did not apply nor was there really any suggestion that the word 'synthetic' did not apply either. Therefore it had really come down to whether the word 'chemicals' was to be used in that strict sense.

Referee: 'There was a conflict, was there not, as to the meaning which should be given to the word "synthetic"?'

In his submission, stated Sir Lionel, there was absolutely no genuine controversy about 'synthetic' at all. The real controversy was over the word 'chemicals'.

If the words of the Act were 'chemical compounds', Sir Lionel said he would agree that the argument of Mr. Graham would have great force. Then it would have to be decided whether the polymers were or were not compounds in the strict sense. 'Compounds' would have had to be defined with great strictness, but he suggested that that was just



exactly what Parliament did not do. Parliament had taken the word 'chemicals' instead of 'compound'.

Hack's dictionary, Sir Lionel recalled, had played a curious part in the case. It had been introduced and put to the complainants' witnesses but it had never been produced for the leading B.o.T. witness, Sir Christopher Ingold. Mr. Johnston and Mr. Graham did not really rely on Hack.

The most interesting piece of evidence as regards what a 'synthetic organic chemical' was in proper connotation in 1921 was provided by the B.o.T. In 1921 the B.o.T. had defined a 'synthetic organic chemical' as including cellulose acetate. That could not be disregarded as a matter of evidence.

Mr. Johnston's argument for the B.o.T. dealing with the comments by Dr. Dreyfus (made in *CHEMICAL AGE*, 19 February 1921), Sir Lionel said, was one of the most cynical arguments that ever been put forward on behalf of any Government department. Mr. Johnston had said that Sir Lionel had put forward the article of Dr. Dreyfus, etc., and had suggested that cellulose acetate was the type of material essentially contemplated by the Safeguarding of Industries Act. Mr. Johnston had said that was completely wrong; maybe they had wanted to get it on to the list, but it did not follow in the least that they would. That was all. He did not add that although, according to his argument, it was quite wrong to suppose that cellulose acetate was ever intended to be in the Act—in fact it was put in in the very first list that was ever produced.

'Here is something, which according to our argument and the evidence upon which we have based it, was clearly and essentially the kind of thing involved in the mischief that the Act was intended to remedy, and you could say in those circumstances it would be reasonable to find it was included in the list'.

### B.o.T. Not 'Tyrannous'

It was stressed by Sir Lionel that he did not want any mistake to be made. He had never suggested that the B.o.T. had been behaving in an improper and tyrannous way and exacting millions of pounds of duty from people wrongfully. Mr. Johnston said it could not be said that the B.o.T. had been acting illegally because the Act had the force of law and had to be regarded as equivalent to the Act of Parliament, but at the same time it could be said that they were wrong. It could not say be said it was illegal, but it could be said to be wrong, which Sir Lionel considered a most extraordinarily cynical argument.

Referee: 'It may be one of the consequences of leaving the administration of an Act to a Government department. It may be that Parliament is at fault if that is the consequence, but it could be a consequence, could it not?'

Sir Lionel: 'It may be, may it not, that it is just that their argument is wrong?'

Referee: 'That is another possibility'.

## C.A.'s Exclusive Report of Tribunal

The tribunal's proceedings have been reported exclusively in *Chemical Age*. No other trade or technical journal or national newspaper has covered the hearing and this paper's extensive reporting, apart from the official transcript, represents the only record of what has been an historic case; the first of its kind in the chemical industry under the Act. A *Chemical Age* feature on the Safeguarding of Industries Act published in February 1921 before the Act reached the statute book was at that time also an 'exclusive' for when a search was made in connection with the recent case, it was found to be the only reference of its kind. This feature extensively quoted at the tribunal, gave the views on the future of U.K. key industries of some prominent chemical manufacturers.

Sir Lionel said he wanted to make it perfectly clear that what he was suggesting was that the attitude of the B.o.T. in this case was wrong.

Referee: It may well be, but let me put my difficulty to you. I do feel difficulty in seeing what is the materiality of this decision of the B.o.T. I accept that there can be criticism, but what is its materiality to the construction of the Act? That is what we have to fasten our minds on'.

The first thing was purely a matter of evidence, stated Sir Lionel. One had to arrive at the meaning of the term in those days. 'The fact that the responsible authority delegated by the Government to make the list did in fact put it on must in fact be evidence, in fact very powerful evidence.

'What he was saying was that there was a substantial degree of authority which said that other things being equal the court would be reluctant to give a meaning to an Act which would have the result of indicating that public and other authorities had been acting improperly or illegally but for the Act, the fact that they could not be challenged. Any construction of that kind would be avoided if there were a proper and reasonable way of construing the Act'.

The referee said he would have thought that the strongest argument on the complainants' side was that there was some evidence as to what the construction was!

Another argument was that all there was to consider was the question of construction of the list. The schedule was a presumption of law, Sir Lionel maintained.

The referee pointed out that 'chemical' on Sir Lionel's construction could mean that 'chemical' had to be construed in other parts of the schedule in the same way and that would mean that chemicals manufactured by fermentation processes could include beer and wine. He was, however, entitled to consider the true rules of construction and what would be the effect of giving a certain meaning to the word 'chemical'.

Regarding beer, Sir Lionel said he had had a very good answer from Sir Christopher who had said there was no analogy between polymers and beer at all. There was no difficulty in saying that when Parliament said 'chemicals' they did not mean 'beer', but it did not follow from that that they did not mean polymers should not be in the list.

The referee suggested that it did not follow that chemicals manufactured by fermentation processes would not include beer on a certain view of the word 'chemicals'.

Sir Lionel said that the things that were described as 'beer' were not described as 'chemicals'.

The argument that there were other things that were bound to come in if polymers came in did not follow. There was no evidence that in 1921 polymers had a separate appellation under which they were dealt with, for example, on export.

Turning to the *CHEMICAL AGE* article in 19 February 1921, Mr. Johnston had indicated that the other case where protection had been demanded was in the case of thorium and rare earths and for incandescent lamps. Mr. Johnston said that cellulose had got on the list and thoriums and rare earths, etc., had not.

Referee: 'To do Mr. Johnston justice in that respect, was he not referring to the schedule to the Act which refers to other rare earth materials? I thought he was'.

Sir Lionel considered that the first list would surely be somewhat relevant. The rare earths were there too. But the curious thing was, stressed Sir Lionel, in the present case, although according to Mr. Johnston they were not within the mischief of the Act they were put in, which did seem to be most extraordinary. There was not only cellulose acetate, there was oxamide.

Referee: 'There are at least six'.

Another companion point in connection with the list was the case of formaldehyde and paraformaldehyde. Paraformaldehyde was, he thought, in the first list. It was what was called an

## 'Fantastic' Position of Monomer being Listed, but not the Polymer

addition polymer, and Sir Christopher had pointed out that it was a convenient way of packaging formaldehyde. It had been said that it ought not to be in. Polymethyl methacrylate could be depolymerised to the monomer, then repolymerised again and that, according to Mr. Raine (see *CHEMICAL AGE*, 15 November, p. 800) was actually done commercially. The two cases were indistinguishable in principle, because if paraformaldehyde was off the list—it was actually on—formaldehyde might just as well be removed. The absurdity of the situation was really found when it was realised that the main use of the acrylate monomer was actually for polymerising. (Dr. Cook's evidence.)

'Therefore if one construes the Act in such a way as to achieve the object of Parliament, unless one applies that principle, one must say that we cannot protect formaldehyde, although that is a chemical, because we cannot put paraformaldehyde on the list and so the result is clear.

'One really has got this position, that where you have got a case where the monomer and the polymer are commercially interchangeable it is a fantastic result if one of them is on and the other is not on'.

### A Raw Material Act

Mr. Graham, Sir Lionel recalled, had said the Act was a raw material Act. He had therefore found it impossible to understand how Dr. Barb could say that the material present in the bottles was not a chemical raw material. Dr. Barb had said that the material was imported for the purpose of being fabricated into manufactures, but that it was not a raw material. The reason why Dr. Barb had found himself in that position was that he had previously been speaking of chemical raw material, and he had been leading up to the point which he (Sir Lionel) did lead up to—that that was a chemical raw material; 'and I am sorry to say this, but he quite clearly appreciated that and said "I will not agree it is a raw material because of course if it is a raw material it is a chemical raw material". Then Mr. Graham, in his final submission, had said this was a raw material and that the polymers in the case were chemical raw materials'.

Referee: 'He did say it is a raw material Act'.

Sir Lionel: 'This being a raw material Act, and this being a chemical raw material—it is not very surprising that he (Dr. Barb) did not want to accept it'.

With regard to cellulose acetate, the point was that it was a most important item in wartime. Sir Christopher remembered that perfectly clearly. Sir Christopher's evidence on cellulose acetate, Sir Lionel said, had not been re-

ferred to but was, he suggested of much better value to the referee than Mr. Graham's submission on it. Attempts had been made to belittle it, but it was not a matter to be belittled. It was the very kind of thing that one wanted to know when one was going to give the construction of the Act.

The evidence that had been given by Sir Christopher on the words 'compounds' and 'synthetic' was reviewed by Sir Lionel (see *CHEMICAL AGE*, 22 November, page 852).

Sir Lionel maintained that the value of 'synthetic organic chemicals' as a phrase in Mr. Graham's definition was absolutely nil.

Referee: 'You mean if it only includes fine chemicals'.

Sir Lionel: '... what were the main things that they were going to cover under this heading of "synthetic organic chemicals". The answer was that that did not mean anything at all, it was nothing to do with it.

### Would Exclude Elements

On the point about the inclusion of elements, Sir Lionel said the words 'synthetic organic' would exclude the elements, because there must be a combination of elements for it to be 'synthetic' and to be 'organic'.

Mr. Johnston said that was accepted; it was the 'fine chemicals', that included things like phosphorus. . . .

Referee: 'That was the point, an element such as phosphorus could be a fine chemical, that was what I thought'.

Mr. Johnston: 'There was evidence that it was'.

Referee: Yes. It was advanced, Sir Lionel, as I understood it, as a possible explanation as to why the draftsman of the schedule had not used the words 'chemical compound'.

Sir Lionel: '... I understand what is said is this, that if the word "compound" had been used, you would still have been able to achieve the purpose that my learned friends say they want to achieve, by referring to fine chemicals as well, and there is no reason why the word "compounds" should not have been used, because the elements would come in under the fine chemicals. . . . The question is this, there may be something which is reasonably expected to be included and yet is an element and not a compound? If that is so, then according to any evidence we have got that would be a fine chemical, and that would be an excellent reason for including the fine chemicals, and also putting in the word "other", because there would be some which you have already covered, some which you have not covered, and the ones which you have not covered would include the elements'.

Sir Lionel said he was merely pointing

out that Mr. Gratwick was saying that the answer to the suggestion that the word 'compounds' could not be in because it would not do the job. It would have been possible to cover the whole thing by saying that reference could be made to fine chemicals.

Referee: 'Even with the expression "all other fine chemicals"?'.

Sir Lionel: 'Yes, because some of them would already be in; some of the synthetic organic chemicals would already be in. Then these other ones, the elements, which you would also want to include: the argument again would be perfectly consistent, and the only result of it all is that it goes to show that Parliament could have achieved its object by using the words "compounds", but it deliberately decided not to use the word, and one can draw it from that'.

One more point that Sir Lionel mentioned was that all the witnesses agreed that the polymers could be identified sufficiently for Customs purposes. His questioning on the Brussels nomenclature was done because some suggestion was made to the effect that those categories were so indefinite that they were not suitable things to make the subject of inclusion in the list. 'The interesting fact is that for the future, for the purpose of all these international agreements and classifications, and possibly free trade area . . . they have all agreed that the definition 39.02 is a perfectly satisfactory one. That seems to show that if the decision were in favour of the complainants the Board of Trade can perfectly well take care of how it is going to be put into the list and define it. They have been a party to this international agreement, presumably advised by competent people, and they have all come to the conclusion that those are perfectly satisfactory definitions, so that you need not be deterred from giving your decision by the fear that there is going to be some difficulty . . .'

### No Deterrent

Referee: 'I should not be, Sir Lionel, I can assure. If I felt as a matter of law, and my colleagues felt as a question of fact, that the decision should be in favour of the complainants, I should never be deterred by possible difficulties of definition.

Sir Lionel: 'No, but I think you would be reassured by knowing of those broader definitions than anything we have got here—you have looked at them. I am sure—they are extremely broad; they are not as bad as the "formaldehyde, whether polymerised or not", they have at any rate treated the polymerised ones as separate, where the Board of Trade in their more virgin innocence seemed to think you could have a category which included polymerised and unpolymerised'.

Finally Sir Lionel said he wanted to mention 'mixtures.' He thought Mr. Graham was unaware of the situation. It was the B.O.T.'s argument that mixtures were out; that had been a matter of controversy for months, even for years. Sir Lionel quoted from the B.O.T. document of 8 August 1948—"the said

articles are mixtures whose innumerable components . . . The fact that they were mixtures had always been relied on strongly by the Board of Trade, who had adopted the position as he understood it that the Act could be evaded by simply importing two things at once. 'I say that that is the most fantastic thing and shows that their whole approach to the matter is wrong.'

Referee: 'It rather ignores the provisions of the Finance Act 1901, does it not?'

Sir Lionel suggested it all depended how these were applied. Reference had been made to section 1(4) but that was not really appropriate to the pure mixture case at all. As he understood it, the polymers in question were single chemicals. Not much had been heard about it in the present case. He did not know whether the B.o.T. were still insisting on it or not—possibly they had been convinced that it was wrong.

It was repeated by Sir Lionel that he had not suggested, and had never had suggested, that the B.o.T. was acting improperly or wrongly in 1921, but he did say that it was a significant comment upon their attitude in the present case that if their argument in this case was right they would in fact have been acting in such a way, for 36 years, that

as a result over the past few years they would have been obtaining hundreds of thousands of pounds in duty which they never ought to have had at all.

Referee: 'As a matter of finance, I would very much doubt that is borne out by any evidence at all, nor by the material provisions of the various statutes to which one has been referred. You might be right, but there is absolutely no evidence of that.'

Sir Lionel: 'It is Sir, in relation to cellulose acetate'.

Referee: 'We have no figures as to . . .'

Sir Lionel: 'We have no figures, but I do not think anybody requires, certainly I do not believe your colleagues would require to be told, the amount of materials involving cellulose acetate that are imported yearly at the present time; it is a pretty large figure.'

Referee: 'Yes, but all I mean, Sir Lionel, is that what we do not know is how much duty would have been exacted if they had not been on the list—not under the schedule, but irrespective of it.'

Sir Lionel: 'Certainly nothing like 33½ per cent. At any rate, there it is. Those are matters which as I say can be reviewed and are going to have to be reviewed in January of next year, and indeed it may even be possible that somebody has thought of them already.'

## Unique System Produces Polyester Resins by Automatic Temperature Control

A NEW polyester resin plant at the Oldbury works of B.I.P. Chemicals Ltd. requires no attention with respect to temperature control from the moment the reactants are charged into the vessels until the finished product is discharged. This is brought about by a unique system of instrumentation which enables 1,000-gall. batches of polyester resin to be produced under fully automatic temperature control from start to finish.

The control system was designed by Hygrotherm Engineering Ltd. in collaboration with Negretti and Zambra Ltd. A programme controller and a slave controller are the principal units of the system. The programme controller compares the required batch temperature, as determined by a cam profile, with the actual batch temperature. The output signal deviates from 9 p.s.i. by

an amount depending on the difference between the actual and required batch temperatures. This output air signal from the programme controller sets the control point of the slave controller.

Next, the slave controller regulates the temperature of the heating medium admitted into the reaction vessel jackets. The output signal of the slave controller depends on the deviation of the heating medium temperature from the temperature corresponding to the signal received from the programme controller.

The control system is working most successfully and batches are controlled to within  $\pm 1^\circ\text{C}$  of the required temperature. The system, it is said, could well be applied to other processes; including those in which it would be necessary to determine the end point for individual batches by other means.

## Synthetic Resin Plant Extensions



Shown here is part of the resin plant at Machen, Mon., of Cray Valley Products Ltd. Extensions now in hand will raise capacity for synthetic resins (alkyds, polyamides, phenolics, Epikote esters, etc.) by 50%. Due for completion by early 1960, they will cost about £100,000

## Import Duty on Sulphate of Ammonia Opposed

AN application for removing the £4-a-ton import duty on sulphate of ammonia has been made to the Board of Trade by the Farmers' and Smallholders' Association, reports Mr. Richard Lamb, chairman of the association.

World prices of sulphate of ammonia had collapsed in the last two years, but home prices had risen, stated Mr. Lamb. British farmers used the equivalent of 1.5 million tons a year. As the British price was £5 a ton above the European price, farmers were overcharged by the fertiliser monopoly 'to the tune of £7.5 million a year'.

Last May, the association had made an application for the removal of the tariff. Although the National Farmers' Union had not supported the application many individual farmers and chairmen of N.F.U. branches had done so. The Farmers' Union of Wales had already written to the Board of Trade in support of the application.

Lowest bulk price for sulphate of ammonia to compound fertiliser manufacturers in Britain was £19 3s. 9d. Bulk supplies on the Continent were offered at £14 6s. a ton.

## Chemicals Exempted from Key Industry Duty

THE Treasury have made an Order exempting the following chemicals from Key Industry Duty until 31 December:

Adenine sulphate; 5-amino-1-naphthol; *mono-tert*butyl-4-hydroxyanisole (a hydroxybenzene ether homologue); copper aceto-arsenite; 4:5-dichlorobenzene-1:3-disulphonamide (a substituted arylsulphonamide); 3:4-dichlorobenzoyl chloride; diethyl ethoxymethylenemalonate; 2:4-dihydroxybenzaldehyde; dimethyl maleate; ethyl benzoylacetate; ethyl 4:4'-dichlorobenzilate.

*n*-Heptoic acid; *n*-heptyl alcohol; *m*-hydroxybenzoic acid; 2-methoxycarbonyl-1-methylvinyl dimethyl phosphate (a methyl ester); 1-phenyl-5-sulphanilamidopyrazole; potassium 4-aminosalicylate; radioactive isotopes, artificial, and compounds; disodium benzene-*m*-disulphonate; sulphaproxyline (a substituted arylsulphonamide); DL-threonine; trichloroacetic acid of a purity not exceeding 95%; 2:3:6-trichlorobenzoic acid; 3:3:5-trimethylcyclohexyl mandelate; zinc sulphite.

The Order which came into operation on 29 November is the Safeguarding of Industries (Exemption) (No. 7) Order, S.I. 1972/1958.

## Duty-free Imports of Certain Organic Products

Under Section 6 of, and the Fourth Schedule to, the new Import Duties Act, 1958, the Treasury can, on the recommendation of the Board of Trade, issue directions that payment of duties chargeable on certain organic products (dyestuffs and intermediates) shall not be required. The Treasury can only take this action if similar articles are not for the time being procurable in the U.K.





★ IN VIEW of the polymer arbitration tribunal's baffling ruling that polymers are not 'synthetic organic chemicals' and therefore not subject to K.I.D. of 33½%, it is interesting to note that the Australian Tariff Board is now considering a request that a dumping duty be imposed on imported Japanese p.v.c. resins. It was the mounting imports of cut-price Japanese and Italian p.v.c. that led the U.K. polymer industry to seek a ruling that polymers should be defined as 'synthetic organic chemicals.'

They can now, of course, take the more circuitous path of approaching the Board of Trade to raise the present import duty to something like rates of competing countries. I gather that so far no action has been taken, either collectively or individually. Of course, no firm, least of all 'giants' like Bakelite, Distillers, I.C.I. or Monsanto, merits protection because it is subject to competition, the life-blood of healthy enterprise. But when these firms are denied entry to the very countries which flood their home market with cheap material, then action is vital, not only in the interests of the industry and its workpeople, but also in the national interest.

If I.C.I. of Australia and New Zealand succeed in their bid to cut out unfair competition, then the Australian Customs would impose a dumping duty which would represent the difference between the export price of p.v.c. and the cost of production of imports, plus an addition not exceeding 20%, plus f.o.b. charges.

★ WITH the start-up of Laporte Chemicals new hydrogen peroxide plant at Baronet Works, Warrington (see CHEMICAL AGE, 29 November, p. 895). I was reminded that apart from the company's large electrolytic plant producing hydrogen peroxide, there is only one other producer in the U.K., Alcock Peroxide of Luton. The research on the new process, which is an organic one, has been carried out at Laporte's research centre at Luton since about 1947.

Demand for hydrogen peroxide has grown steadily over the past decade, and rapidly during the past few years with the development of rockets.

In the U.S. there are five producers, Pennsalt, Food Machinery Corporation Becco division, E.I. Du Pont de Nemours and Co., Columbia Southern and Shell. The last named started up a plant in January this year, which produces as by-product glycerine. In Europe, Germany has four plants, France and Italy have two each with a third shortly to be completed in France. Belgium,

Sweden, Switzerland and Yugoslavia each has one plant. There are believed to be several in the U.S.S.R.

The largest plants are in Germany, France and Italy and all use the electrolytic process. Solvay, however, are understood to be planning an organic plant for Belgium and probably for France and Germany as well.

Mr. L. P. O'Brien, chairman of Laporte, told me last week that the price of hydrogen peroxide was the same as 20 years ago. The rapidly rising cost of electricity, however, inhibited any further economies in price. The new organic process, the company consider, will prove much more economic. At the same time, with interest centred on H.T.P. as a fuel, new fields will be entered which have hitherto been barred on economic grounds.

★ IN A new catalogue just issued by Quickfit and Quartz Ltd., packaging rather than the equipment itself makes news. The glassware is standard but it is assembled into units which are complete in themselves. These units are cheaper to buy than the individual pieces which go to make them up and replacement is also cheaper. Packaging of these assemblies is neat and serves as storage space in the laboratory for drawers and cupboards can take the complete unit. Breakages in transit should be considerably reduced. By comparison with old methods of packing, this assembly occupies only about half the volume. No extra material, such as wood wool, etc., is needed in transit.

★ MORE details of the U.S.S.R.'s plan to spend over the next seven years a total of 100,000 million roubles on its chemical industry are reported from Moscow. In that period some 250 new plants will, it is said, be set up. Of this investment a large proportion will be spent on expanding the country's plastics industry; an example of the ambitious steps aimed for here is the raising of the polythene production to an annual 400,000 metric tons by 1965.

Production of raw materials, particularly mineral oil, in sufficient quantity to meet this large increase in plastics production, as well as a planned rise in the manufacture of synthetic rubber by 250 per cent, is also foreseen. Suppliers of plant for the plastics and synthetic fibre industries' expansion have been named by the Russians as the U.S., the U.K., Western and Eastern Germany and Czechoslovakia. Technoprom, the State body for the import of industrial plant, has been in touch with British

firms regarding plant and other chemical and petroleum equipment and has issued detailed instructions for what its requirements would be.

Soviet scientists are busy with work on the production of ethylene, propylene and monomers from oil and natural gas, and new processes for the production of isoprene are being developed. Research is also being carried out on the production of nylon from an ethylene base; the Russians say that owing to the cheapness of such a process, such a nylon might well become a serious competitor on world markets with conventional nylon 'in a few years.'

★ POLYTHENE—sometimes known as polyethylene, less frequently as polymethylene—is always in the news these days. Recently I have talked about its use in hula-hoops and for stencilling. Now comes another new use, but in the far-removed field of ballistics.

The U.S. Naval Ordnance Laboratory has developed triangular cartridge cases in linear polythene for a new pistol of revolutionary design. All other parts of the ammunition are conventional but the pistol uses the split-chamber system, cutting out the forward motion of ramming a cartridge case into the firing chamber.

Elasticity of the polythene allows the case to expand under high pressures without bursting and provides a superior gas seal at all cylinder joints. New styrene-derivative 105 mm. shell cases, also designed by N.O.L., have withstood temperatures of up to 4,300°F and pressures up to 35,000 p.s.i. of actual firings.

★ WHILE most of the entrants for the Dip. Tech. sandwich course in industrial chemistry at the Northampton College of Advanced Technology, London E.C.1, are directly sponsored by the chemical industry, increasing inquiries are coming from students who have grants from local education authorities and who are available as trainee chemists with selected firms for the four six-month industrial periods alternating with the college periods.

The college believes that these students can form a valuable source of recruitment to the scientific and technical strength of a firm. An intake every six months ensures a continued even loading of the firms' laboratories, research and development section, etc. The college will receive its third intake of students for the course in February next, followed by a fourth in September.

Firms considering recruitment from this source are invited to write to Dr. J. Leicester, head of the department of applied chemistry at the college, St. John Street, London E.C.1, for further particulars.

*Alembic*

## ANALYSTS DISCUSS IDENTIFICATION OF NEW PERMITTED FOOD COLOURS

WHEN the Food Standards Committee of the Ministry issued its Report on Colouring Matters in 1954 (1), followed by a Supplementary Report in 1955 (2), recommending introduction of legislation restricting colours used in food to those specified in a permitted list, it appeared that yet another problem was to be set for food chemists and public analysts in particular. In order to be ready with an answer to this problem the Association of Public Analysts, in 1956, set up a committee to study the subject.

Much of the lecture by Mr. P. S. Hall (Bernard Dyer and Partners) on 'The identification of the new permitted food colours', given at a recent meeting of the Midlands Section of the Society for Analytical Chemistry at Nottingham and presented also at the S.A.C.—Association of Public Analysts symposium on 'Food analysis' in London on Wednesday this week, was based on recommendations of this committee viewed in the light of later experience.

When the Colouring Matter in Food Regulations (3) were published in 1957 a list of 30 coal tar colours was included, 28 of them water soluble. The problem which they set was briefly examined in Mr. Hall's lecture.

### Water Soluble Colours

Considering first the water soluble colours, permitted dyes could be divided into five classes, namely, azo, xanthene, triphenylmethane, nitro and indigoid. All were acidic and there were, in fact, no basic dyes permitted by the Regulations. From the public analyst's point of view it was required to determine whether the colour was permitted, or not, but since the majority of non-permitted dyes fell into these same five classes, a mere identification of chemical class was insufficient. Apparently, the only satisfactory way was by positive identification of the colour concerned.

From methods published in the literature, it was found that the A.O.A.C. (4) described a technique involving solvent extraction. This appeared to be unsatisfactory since it was purely empirical and there could be no forecast of the way in which any given dye would behave. Chemical tests had been described by Greene (5) and by Clayton (6) with special reference to textile dyes. These, it was thought, might be of some use since they gave an idea of the chemical type of an unknown dye but, of course, they were of no use for positive identification. It seemed that the methods most likely to be of use would be physical

methods, such as paper chromatography and spectrophotometry, rather than chemical methods. In Mr. Hall's laboratory, paper chromatography had been used for some time, in a rather haphazard way, for identification of colours, and another member of the association's committee (Dixon) had studied the problem in a more systematic manner.

The committee decided to study this approach to the problem in detail and found that it was possible, at least, to obtain preliminary identification of unknown colour by paper chromatography. A number of solvents were tried and six of these were chosen as being most satisfactory and sufficient for most purposes. The A.O.A.C. had been working on similar lines and two papers were published by Tilden (7, 8) giving a list of some 27 solvents. None gave entirely satisfactory results in the hands of Mr. Hall and his colleagues.

### 'Not Critical Enough'

Apparatus used for chromatography did not appear to be too critical. For initial sorting the C.R.L./I paper of Whatmans, as described by Hunt, North and Wells (9), was very convenient. Alternatively, a plain sheet with a vertical slot cut at either end could be coiled in a cylinder in the same way as the C.R.L./I. The coiled paper was placed in a litre beaker with the solvent in the bottom of the beaker and the whole covered with a clock glass. In order to separate dyes having very similar Rf values a longer run was sometimes desirable, in which case a Shandon tank giving a run of up to 18 inches was used.

A technique which Mr. Hall favoured, and which had the advantage of simplicity, was a modification of the disc method of Rutter (10). The apparatus consisted of two sheets of glass, approximately 8 inches square, one of these having a small hole bored through the centre. In operation a circle about  $\frac{1}{2}$  inch in diameter was drawn in the middle of a filter paper circle and this was divided into a number of arcs. A tail, approximately  $\frac{1}{4}$  inch in width, was cut from the edge of the filter paper to the centre and the dyes applied, each along a section of the circle. After the dyes had dried, the tail of the filter paper was passed through the hole in the centre of the lower glass plate and so arranged as to dip into the solvent contained in a Petri dish. The plain glass plate was placed on top of the filter paper. With this technique, it was possible to differentiate between dyes having

very similar Rf values. If the unknown was spotted with the known dye on the arc on either side of it, a continuous circle was produced after development if the dyes were identical. Rf values for all permitted dyes with the various solvents were then given by Mr. Hall. These values should, however, only be used for guidance, since experience showed that Rf values were quite unreliable as a means of identification. The only reliable method was to run unknown and known dyes alongside one another on the same paper.

In practice, the unknown was put on a chromatogram alongside known dyes of similar shade. Choice of solvent was largely a matter of experience. Frequently the first chromatogram would indicate the identity of the unknown. A second chromatogram with a different solvent would usually confirm it. At least half of the colours used in food were, in fact, a mixture generally of two, sometimes of three, dyes. Often it was unnecessary to separate these dyes prior to chromatography but occasionally it was desirable.

Separation of such mixtures had been described by the A.O.A.C. (11), use being made of a column of cellulose from which the dyes were eluted with sodium chloride solution of gradually diminishing concentration. Mr. Hall's panel, while achieving some success with this technique, found that the bands of the individual dyes frequently overlapped, thus requiring to be passed through the column a second time. An improved technique was devised by Dixon employing, in fact, large scale paper chromatography, the mixed dye being painted in a strip along the starting line of the chromatogram and developed with a suitable solvent. In most cases clear separation of the dyes was obtained. The individual colours could be cut out from the chromatogram and extracted with water. Sufficient dye was usually obtained in this way for subsequent tests.

### Useful Confirmation

Spectrophotometry provided useful confirmation of the evidence obtained by paper chromatography, and three members of Mr. Hall's committee had plotted and checked the ultra violet and visible absorption spectra of all permitted colours in 0.1N acid, in 0.1N alkali and in neutral solution. A technique which was sometimes useful, with only a limited amount of dye available, was to cut out the coloured zone shown on the chromatogram, moisten it with a little glycerin and insert it in the cell carrier of the spectrophotometer. The absorption spectrum was measured through the paper; a similar unstained paper, treated in the same way, was used as reference.

Identification involved, therefore, comparison of the properties of the unknown with specimens of known dyes. This was fairly simple when it was known that one was dealing with one of the 28 permitted water soluble colours, but if the

unknown were not found to be a permitted colour it would be a very tedious business to make chromatographic comparison with every colour of similar shade used in food. A procedure which would eliminate some of the possibilities was needed.

Of the techniques mentioned previously, the scheme of Clayton was chosen for further examination. This classification scheme had the advantage that it was not empirical; in other words one could forecast, with some confidence, how a given dye would react. Clayton's original tables had required some editing and one or two amendments had also been made. The revised scheme was shown in some detail by Mr. Hall. Reagents referred to were those used by Clayton, and the whole scheme was to be read in conjunction with Clayton's comments (6) to his original scheme. By means of these tests the possibilities were limited and one did then know, at least, in which chemical class to search for the unknown dye.

To sum up then, it was possible to be fairly confident of identifying an unknown dye, provided that one had a known specimen of that dye with which to compare it. This was, however, useless unless the colour could first be extracted from the food.

### Extraction of Dyes

Little help was obtained from the literature on the question of extraction of colouring matter from food. The technique usually employed was to dye defatted white wool from a weakly acidulated solution and subsequently strip the dye with dilute ammonia solution. In the case of basic dyes, this was not too satisfactory although some colour was extracted. Lack of information on this subject might be due, in part, to the range of products which might contain artificial colour, each one requiring a different approach. However, a wool dyeing technique appeared to be the only one likely to prove satisfactory and this was concentrated on exclusively.

Before attempting to extract the dye, a very useful test was to view the sample in ultra violet light. The xanthene dyes, and especially the Rhodamines (Rhodamine B was one of the more popular of the non-permitted dyes), showed a characteristic intense fluorescence.

For the purpose of extraction dyestuffs might be divided into two classes, acidic and basic, and foodstuffs might be divided into those which gave a solution with water, such as jam, table jellies, soft drinks, and those which could not be made directly into a solution for dyeing a piece of wool, such as custard powder, coloured starchy products generally, meat and fish products. The latter had to be extracted with a suitable solvent to remove the colour. 1% ammonia was suitable for acidic dyes and 5% acetic acid for basic dyes. There appeared to be no way of telling whether the colour was acidic or basic at this stage, so both solvents had to be tried. For a material such as custard powder, heating was clearly not permissible; and the colour was extracted

with cold 1% ammonia. The powder was shaken with the extracting liquid, allowed to settle overnight or centrifuged, then the clear solution of the colour poured off.

Ammonia extraction was liable to cause trouble when applied to protein materials, more especially to fish. It appeared to extract a considerable amount of albuminous matter which, when the solution was acidified in order to dye the wool, was precipitated and, of course, preferentially extracted the dye, leaving none to be extracted by the wool. Acetone was frequently of some use in these cases. The sample was first defatted with ether, then extracted with acetone containing about 20% of water. Under these conditions the dye was extracted fairly well and without too much interfering matter. The acetone was subsequently removed by evaporation and the residue taken up in water.

Having obtained an aqueous solution, the test for basic dyes, described earlier, could be applied. If they were absent, the solution was acidified with acetic acid and sufficient defatted white wool put in the boiling solution just to extract all the colour. The dyed wool was removed from the liquor, washed under the tap, the dye stripped from the wool by boiling with 1% ammonia, and the solution so obtained evaporated to dryness. The residue was taken up in a few drops of water, so providing the test solution for application to the chromatogram.

There were only two permitted oil-soluble colours. Both of these were azo compounds, one a pyrazolone derivative,

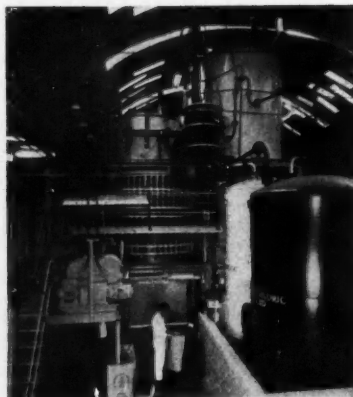
the other a resorcinol derivative. The latter being phenolic could, of course, be extracted from ethereal solution by dilute alkali. A third type of colour which might be encountered was that formed by coupling a diazonium salt with an aromatic amine.

For routine work, the simplest procedure was to separate the colour by passing a solution of the fat in petroleum ether through a column of activated alumina. The adsorbed colour was eluted with petrol containing increasing proportions of ether. The dye fraction was collected separately and evaporated to small bulk. Comparison of the separated colour with known dyes by paper chromatography provided a preliminary identification. The paper had first to be impregnated with liquid paraffin by dipping in a 5% solution of liquid paraffin in ether and drying. The disc technique, described already, was found to be the most satisfactory. Details of suitable solvents were given. Confirmatory evidence was once again obtained by comparison of absorption spectra.

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### Price's New Plant



The new soda-saponification plant recently commissioned by Price's (Bromborough) Ltd., enables them to offer 12-hydroxystearic acid (hardened castor oil fatty acids), technical ricinoleic acid and tung oil fatty acids in commercial quantity from their Merseyside works. (See also 'Chemical Age,' 22 November, p. 862.)

### New Laboratory for Anderson's

James Anderson and Co. (Colours) Ltd. have been granted planning approval for the erection of a new three-storey laboratory block at their Portland Road, Hawkhead, Paisley, site.

### Instrumentation In Plant Design

INSTRUMENTATION and computation in process development and plant design is the title of a joint symposium to be held at the Central Hall, London SW1, from 11 to 13 May next. It is to be organised by the Institution of Chemical Engineers, 16 Belgrave Square, London SW1, in conjunction with the Society of Instrument Technology and the British Computer Society.

Session titles will be: Improving the efficiency of existing processes; Design of new processes; Application of on-line computers; Recent developments in instruments, on-line computers, and computers for design; Use of computer techniques in large and small companies.

### Reprints of C.A. Plant Survey

The six-page report of new U.K. chemical plant projects published exclusively in *Chemical Age*, 22 November has been reprinted. Copies are available, free of charge, on application to the Editor at 154 Fleet Street, London E.C.4.



## Analytical Review

# Direct Non-Aqueous Determination of Uranium

MUCH interest is currently centred on the determination of uranium following its separation from other materials in aqueous solution by means of extraction into a water immiscible organic solvent. Several solvent systems may be used to extract the uranium, e.g. diethyl ether and tributyl phosphate (TBP) for uranium VI nitrate, and many attempts have been made to determine the uranium *directly* in the organic extract. Such procedures are usually time saving and where they are successful they avoid extra operations, such as repartition into an aqueous solution, which increase the hazards of loss of material and contamination by undesirable substances.

The general approach so far has been to utilise physico-chemical procedures which do not interfere with the chemical equilibrium in the organic extract, e.g. spectrophotometry and X-ray methods, but electrochemical procedures, e.g. polarography, have also been applied. However, the trend until quite recently has favoured aqueous methods for the end-determination. Such methods have been applied after evaporation of the organic solvent or re-distribution into an aqueous phase.

Dibenzoylmethane is an excellent reagent for the determination of uranium VI and the coloured complex may be extracted into a wide range of organic solvents. Consequently most of the recent procedures for direct analysis of uranium in organic extracts have made use of this reagent.

## Methods of Extraction

In one procedure TBP (1 part) and *iso*-octane (9 parts) have been used to extract uranium from a solution salted with aluminium nitrate, and the dibenzoyl methane reagent was applied to the extract; pyridine was used as a buffer and aqueous acetone as diluent<sup>1</sup>. Another method uses tributyl phosphate (7 parts) and ethyl acetate (13 parts) as extraction system and applies the reagent to the neutralised extract after it has been well washed with 5 N HNO<sub>3</sub> or 7.5 N HCl<sup>2</sup>. Others have used methyl *iso*-butyl ketone as solvent for extraction of the uranium from a solution salted with aluminium chloride and have developed the colour with ammonium thiocyanate in the extract diluted with butyl cellosolve and water<sup>3</sup>. More simply, uranium has been extracted with ethyl acetate and the colour developed with dibenzoylmethane after dilution with aqueous ethanol<sup>4</sup>.

The most recent dibenzoylmethane method<sup>5</sup> to be reported uses 0.1 M tri-n-octyl phosphine oxide (TOPO) in cyclo-

hexane as solvent for extraction of the uranium from 1-10 M HCl or 1-4 M HNO<sub>3</sub>. The yellow dibenzoylmethane complex is developed in an aliquot of the extract after suitable dilution with ethanol in the presence of added pyridine. A recovery of greater than 99% is obtained when 9.4 mg. of uranium VI are subjected to extraction from the

By

T. S. West, Ph.D.

In this article Dr. West reviews two recently published methods in which uranium VI is separated from other ions by solvent extraction procedures and is determined directly in the extract by application of non-aqueous absorptiometric technique

above-mentioned acid solutions with 0.5 millimole of TOPO in cyclohexane at an aqueous/organic phase ratio of 1. Similar recoveries are also reported for microgramme amounts of uranium using the same amount (0.5 millimole) of reagent and the same phase ratio. The colour developed by dibenzoylmethane under the selected conditions is rapidly stabilised and exhibits unchanged absorbance for a period of at least five days. The amount of reagent used appears to require careful control since it has an appreciable absorption at the wavelength at which the uranium chelate is measured.

The interference of iron (III), zirconium, thorium and titanium is conveniently reduced by addition of fluoride to the aqueous solution before attempting extraction, but this control is not possible if moderate amounts of aluminium or beryllium are present. High concentrations of fluoride can, however, lead to retention of uranium VI itself in the aqueous phase. The interference of iron III, chromium and vanadium may be eliminated by selective reduction with sodium bisulphite or hydroxylamine sulphate. The addition of EDTA is not effective in suppressing interference by metal ions because of the high acidity employed in the extraction. Other chelating acids behave similarly. Phosphate and fluoride interferences are overcome by addition of Al(NO<sub>3</sub>)<sub>3</sub>, but chloride cannot be tolerated because it promotes extraction of iron III, etc., by the solvent system. Oxalate also appears to

interfere seriously. The working range is from 20-3,000 µg. of uranium in the original solution and the standard deviation is  $\pm 2\%$ . The method appears to be an easy one to operate in that close control of acidity or of time of development is unnecessary. In addition there is no need to wash the organic extract or to use salting out agents. However, as the authors themselves point out, the selectivity is not as high as that of some other procedures.

Another paper in the same journal<sup>6</sup> reports on the problem. In this instance the uranium VI is extracted as uranyl nitrate by 9% TBP in chloroform over a wide range of HNO<sub>3</sub> concentrations and the colour is developed directly in the organic phase with 1-(2-pyridylazo)-2-naphthol (PAN) in the presence of pyridine. The selectivity of this procedure is improved by employing EDTA to prevent the extraction of zirconium and manganese II, both of which form coloured chelates with PAN in the organic phase and it also suppresses the troublesome precipitation of bismuth in the aqueous phase. Cerium IV which extracts with TBP from a nitrate medium and reacts with PAN in the extract, can be rendered harmless by reduction with excess of ferrous sulphate, and thorium is precipitated by means of oxalate before extraction. Fluoride is ineffective in removing interferences in this case and EDTA is successful because the extraction is carried out in only faintly acid (methyl red) solution where the fluoride complexes are hydrolysed, but EDTA chelates are stable.

This method is more selective than that described above and appears to be equally accurate, i.e. 99% recovery with a precision of  $\pm 2\%$ . The colour has attained maximum value within 15 minutes and is stable for at least 24 hours. On the other hand the range of the method, 40-400 µg., is somewhat more restricted and the use of a salting-out reagent, repeated extraction and the washing out of the extracts is required.

In the same publication, papers from two other sources describe the separation of uranium VI from rare earths<sup>7</sup> and from bismuth<sup>8</sup>. Both these sources prefer to separate the uranium VI by absorbing it as its sulphate on a quaternary ammonium ion-exchange resin (Dowex 1  $\times$  8 and 10).

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## Gifts to Leeds University

Among gifts to the Leeds University is £500 from the Shell Group to support postgraduate research in the department of inorganic and structural chemistry. Unilever have given £300 a year for three years to the department of physical chemistry.

## ECONOMICS OF ATOMIC REACTORS FOR CHEMICAL INDUSTRY DISCUSSED BY I.C.I. ENGINEER

**P**ROSPECTS of the chemical industry utilising large quantities of steam at fairly low pressure produced by atomic reactors was discussed by Mr. G. A. J. Begg, division engineering director, I.C.I., Alkali Division, in the 'Symposium on Fuel and Power' organised recently by the Manchester Joint Research Council. Mr. Begg's paper was entitled 'Future trends in the use of fuel and power in the chemical industry'. This and other papers presented at the symposium are to be published by the Council.

Although it might be thought that atomic reactors would be particularly suitable for use in the chemical industry, this was not the case at present. The gas-cooled reactor of the type used at Calder Hall and the power stations now being built, used natural uranium as a fuel and the critical size was consequently very large. To make adequate use of the enormous capital cost, output had to be very large, equivalent to an output of say 100 MW of electricity.

Such a plant was only economic when its full capacity could be utilised and even then only if capital charges were low, as in the electricity generating industry. In contrast, extensions in the chemical industry were relatively small and capital charges high. If a gas-cooled reactor were installed in an existing chemical works it would only be partially loaded for a long time or a corresponding amount of conventional plant would have to be shut down.

### Could be Sited Near Atomic Station

There was still the possibility of building a large continuous chemical factory and a nuclear power station close together so that steam as well as power could be sold to the works. The steady demand of the factory would then help ensure full load conditions on the reactor. That might well be the decisive factor in selecting a site for a new plant, although for safety reasons nuclear power stations were built in places remote from industry.

Water-cooled reactors, with a much smaller critical size than the Calder Hall type, were becoming available and were much better suited to small outputs. However, at present the cost of fuel enrichment and fuel element fabrication was high and those reactors could not compete economically with conventional plant.

The study of a project for the installation of a water-cooled nuclear reactor to serve an existing group of factories showed that the capital cost of the nuclear plant would be about £1.5 million for 170,000 lb./hr. of steam. The proposed reactor circuit is illustrated.

The economic picture might change when more advanced types of reactor became available in the next few years.

Experience would also enable more reliable estimates to be made of the useful working life of reactors. By about 1970 industry might begin to find nuclear steam raising economically attractive. It was possible that research reactors would be installed earlier by companies interested in producing materials for use in atomic stations.

Mr. Begg spoke at length about conventional means of using power and steam in the chemical industry. He believed that the trend for process steam would be towards greater use of combined steam/power plants. He also thought that processes needing temperatures of above 400°C would continue to use direct firing or one of the high temperature media, such as Dowtherm or high temperature salts; economy would result from recovery of heat from waste gases. Processes needing lower temperatures might be converted from direct firing to steam with consequent additional back-pressure power generation.

Increased mechanisation and improved process efficiency would mean continual increase of steam pressure. This would, however, be limited by capital cost, special materials, feed water and size of plant. It was unlikely that pressures in excess of 1,600 p.s.i. would become common in this country. Chief factors governing the choice of boiler pressures were: steam-power balance; quantity of process steam required; nature of boiler feed water, permissible live steam temperature. In Germany several plants worked at pressures of 2,000 p.s.i. and above with correspondingly high temperatures, under circumstances where such conditions would be difficult to justify in England.

Unless oil prices rose substantially, oil would be used increasingly to fire small boilers and furnaces, for oil-firing was often preferred to coal for tricky or high temperature processes. Oil-firing gave

cheaper steam on small boilers even when its price was 10% higher. There might well be an increase in the use of oil engines.

In processes where the heat of the fuel was transferred at high temperature, such as roasting furnaces, improvements were likely to be achieved only by improvements in the process reactions themselves, by bringing the actual quantity of heat transferred in the down to a figure close to the theoretical thermochemical quantity by limitation of stray losses through improved insulation of plant and buildings and by recovering low temperature heat from effluent gases. Improvements of this nature were, however, usually limited by capital cost. A reduction in the cost of insulating materials would, for instance, result in substantial saving in heat.

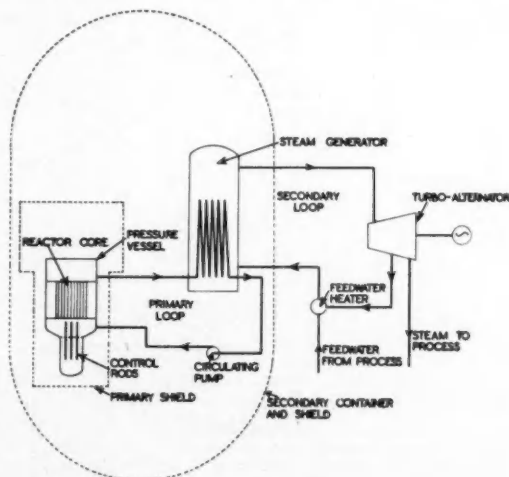
So far as processes requiring heat at below 200°C were concerned there was a trend towards the use of steam instead of direct firing. The advantage was that heat could be released at the highest possible temperature in the furnace of a steam boiler working at high pressure. That steam was then passed through a suitable turbo-generator and finally passed into the process in the normal manner, having generated some useful electricity en route. The process was frequently easy to control.

Schemes of that kind, involving appreciable savings in fuel, often had to be turned down because the return on the additional capital was unacceptable.

Mr. Begg said that the process industries accounted for about one-seventh of the nation's coal consumption, say a total of 30 million tons out of 220 million tons per year. That compared with the Central Electricity Generating Board's annual consumption of about 47 million tons.

### Will

CAPTAIN A. H. COWAP, who joined Brunner Mond and Co. at the age of 13, becoming chief engineer for Synthetic Ammonia and Nitrates, Ltd., Billingham, and who was for 30 years a director of the Power-Gas Corporation, Stockton, left £87,486 net.



Circuit of water-cooled nuclear reactor proposed for serving an existing group of factories

# AUTUMN GAS RESEARCH MEETING-2

## Liquid Reagents for the Final Removal of Hydrogen Sulphide from Gases

TWO established processes for the liquid purification of gas are in use but both have objectionable features; the absorption-desorption processes do not give gas of statutory purity, and the oxidation-reduction methods produce undesirable liquid effluents. In a third method, the reagent absorbs  $H_2S$  to form an insoluble metal sulphide. W.B.S. Newling, London Research Station, The Gas Council considered that some such process might be successfully developed using zinc or copper reagents.

The process had to be thermodynamically capable of lowering  $H_2S$  content of the gas to 0.1 p.p.m. Attempts were therefore made to develop a process using acid copper sulphate solution for the purification of coal gas but the resulting copper sulphide floated and at the same time, the rate of absorption was very low. Another reagent considered was ammoniacal zinc sulphate but unless ammonia were imported into the process, its use would be restricted to gases containing an excess of ammonia over the hydrogen sulphide plus the hydrogen cyanide.

Where large quantities of zinc or copper sulphide are likely to be formed, regeneration of the wash liquor by first roasting the sulphide to the oxide or sulphate would have to be considered and the roast product could then be used to prepare the regenerated liquid reagent for recirculation. Even with modern techniques of continuous-flow roasting in a fluidised bed, the regeneration stages would be a formidable undertaking, and for this reason, the author did not consider it economic to study the process even on a pilot-plant scale.

### Final Purification Method

For some time the North Thames Gas Board had been carrying out final purification using zinc acetate solution to wash the mixed gas, but the reagent was expensive and released acetic acid. At Bromley an important discovery was that the addition of a small proportion of zinc acetate to the soda solution in the washer cooler greatly improved its performance for removing traces of  $H_2S$ .

The zinc acetate used is a suspension of basic zinc carbonate in a solution of sodium carbonate and bicarbonate, with sodium acetate present. Zinc sulphate can be used instead of the expensive acetate, so that sodium sulphate is the inert component of the solution. This reagent was studied at Fulham where rapid absorption of  $H_2S$  was found to depend on the presence of hydrogen cyanide in the gas, but it must not be present in excess. Five works in the North Thames area are already using zinc sulphate and excess sodium carbonate for final purification and a plan is well advanced for the installation of a washer

designed for use with the slurry reagent.

The study of final purification at Fulham has now resolved itself into a search for reagents capable of absorbing  $H_2S$  rapidly from water gas and from other gases containing no hydrogen cyanide but rich in carbon dioxide.

Alkaline copper reagents absorb  $H_2S$  satisfactorily but they have no advantages over the corresponding zinc reagents. Research was concentrated on the discovery of additives having the advantages of cyanide but none of the disadvantages. Although the additives proved to be expensive organic reagents they could be used without wastage. The most promising reagents developed were solutions made from zinc oxide, sodium carbonate and glycine.

### Choice of Reagent

These reagents had the following advantages: Glycine was as effective as cyanide in alkaline zinc slurry reagents but unlike cyanide it could be present in excess without loss of purification performance. The reagent solution could be used for purifying coal gas, and when it is fully dissolved, virtually no reactive zinc has to be rejected with the zinc sulphide product. Utilisation of the zinc always approached 100% and the reserve of the dissolved, rapidly reacting zinc compounds present in the washer was very much greater than with the slurry reagents. Purification performance would not be greatly affected if 90% of this reserve were suddenly used up by a temporary increase in  $H_2S$  concentration in the gas entering the washer. Because of the absence of other solid zinc compounds, the zinc sulphide could be removed continuously from the washer, and zinc sulphide suspensions caused much less fouling of a board-packing than did zinc carbonate slurries.

Final purification of gases by washing can therefore be carried out by these three practical reagents:

- (1) Original slurry reagent made from zinc sulphate and sodium carbonate;
- (2) Improved slurry reagent made from zinc hydroxide and sodium carbonate;
- (3) Reagent solution made from zinc oxide, sodium carbonate and glycine.

**Alkaline slurry reagents:** In practice, the reagent has been prepared by mixing zinc sulphate solution with excess sodium carbonate solution. At Bromley, it was made by adding 500 lb. of zinc sulphate heptahydrate in 500 gall. of water to 3,000 lb. of anhydrous sodium carbonate in 14,500 gall. of water.

The reagent approaches equilibrium with components of the gas other than  $H_2S$ ; most fuel gases contain enough carbon dioxide to convert most of the soda to

bicarbonate, giving pH values of 8.0 to 8.5. Coal gas downstream of the oxide purifiers also contains several grains of hydrogen cyanide per 100 cu. ft.; some of this is absorbed by the reagent so that at the steady state attained, some solid zinc cyanide may be present in addition to dissolved zinc in the form of anionic cyanide complexes. This solid cyanide as well as the hydroxide, basic carbonate, and dissolved zinc, is all available for the removal of  $H_2S$  by conversion to insoluble zinc sulphide.

Hydrogen cyanide has a marked effect in improving the rate of absorption which is associated with a considerable increase in the concentration of dissolved zinc compounds. This concentration may be quickly used up and it is important that it should be continuously replenished by keeping the solid compounds dispersed uniformly in the solution.

$CO_2$ in gas, %	...	0	2.5	7.0
pH	...	11.4	8.35	8.05
$H_2S$ not removed, %	...	0.2	1.0	4.0
Gas-phase resistance, units	...	1	1	1
Liquid-phase resistance, units	...	0	0.4	1.0
Washer size of 0.2% slip, units	...	1	1.4	2.0

Concentration of free cyanide in solution must be very low while there is any basic carbonate present otherwise the zinc ion concentration is suppressed and almost all the available zinc is converted to the full zinc cyanide, which reacts relatively slowly with  $H_2S$ .

### Effect of Alkalinity

**Effect of alkalinity and cyanide content:** In the absence of HCN alkalinity as measured by pH is the factor with most effect on the rate of absorption of  $H_2S$ , and the pH depends mainly on  $CO_2$  content of the gas which cannot be altered very easily. Table I illustrates the effect of carbon dioxide concentration in a series of comparative tests for the partial removal of  $H_2S$ , initially 160 p.p.m., from gases free from HCN and differing only in  $CO_2$  content. The results in the first column are as good as with fresh caustic soda.

The effect of cyanide may be illustrated by comparison with the middle column of the table. Four grains HCN per 100 cu. ft reduced the slip of  $H_2S$  to 0.3%, the washer size for 0.2% slip being only about 1.1 units. Addition of enough NaCN to bring all the available zinc into solution increased the slip to 10% and the required washer size to 2.8 units.

For the final purification of coal gas a large area of gas-liquid interface is the main requirement; board-packed towers are a suitable form of washer.

**Multi-stage washing with slurries:** For use with slurry reagents a board-packed tower may conveniently have a small, shaped liquor well at the bottom with a sloping base so that all solid particles can pass freely to the lowest point and thence to a recirculation pump. A large pilot plant comprising a series of four such washers is designed for washing 0.6 million cu. ft. per day of coal gas or water gas,



## Gas Research Meeting

using the reagent made from zinc sulphate and sodium carbonate.

The 18-in.-bore board-packed towers are each arranged for countercurrent flow, the liquor being recirculated from a cone-based well by a centrifugal pump through a rotameter to trough distributors above the packing. The liquor wells are interconnected below liquor level by sloping pipes arranged for downward transfer of liquor from stage to stage. In large plant the sloping transfer connections would be made to the side of the wells; any liquor displaced down them would be of less than average solids content. No excess of solids can then accumulate in any one stage; actually there is some tendency for the solids to move into the early gas-taker stage in which most zinc is consumed.

Make-up liquor is supplied by three metering pumps for clear liquids, respectively, concentrated zinc sulphate solution, concentrated sodium carbonate solution and water in the form of steam condensate.

Multi-stage operation would only be justified for relatively high concentrations of  $H_2S$  of the order of 20 p.p.m. because the reagent cost would be important and a high percentage of zinc utilisation would be necessary. The system was in fact devised to purify gas normally containing 10 p.p.m.  $H_2S$ , but liable to contain up to 160 p.p.m. during rare emergencies. For this application, oxide purifiers were found to be more economic.

### Inorganic Reagents

**Reagents for purifying cyanide-free gases:** Given conventional conditions, the packed height required in a washer for a given final purification duty would be at least three times as great as for reformed refinery gas containing 7%  $CO_2$  as for coal gas, when using the alkaline zinc slurry reagents. With coal gas, the resistance to the absorption of  $H_2S$  is 10 times less in the liquid phase. Washer size for the reformed refinery gas could be much reduced by adding NaCN to the liquor, but it is poisonous and expensive.

Other inorganic reagents which improve absorption of  $H_2S$  are sodium borate and ammonia, but ammonia is too volatile to be a practical additive. Ammoniacal zinc reagents could be considered only for the purification of coal gas before ammonia removal by washing. The only other effective additives were organic compounds. These increase the proportion of metal in solution, and the performance at a given pH value depends mainly on the concentration of dissolved metal. On reaction with  $H_2S$ , the dissolved organo-metal complex yields the insoluble metal sulphide, leaving the organic additive free in the solution. Dissolved metal concentration is replenished by bringing clear solution, from the separation of the sulphide, into contact with the solid oxide, hydroxide or basic carbonate of the metal.

**Liquid-phase resistance:** The size of washer required for a given duty varies greatly with liquid-phase resistance. With-

out product revenue, the economic advantage of a liquid-purification process over oxide purifiers might be slight but if a large washer were needed, there might be no such advantage. For this reason study was concentrated on systems offering low or moderate liquid-phase resistances not exceeding the gas-phase resistance, so that no more than two minimum washers were needed.

**Falling-film tube apparatus:** Only with some copper reagents or at high concentrations of  $H_2S$  was any difficulty experienced with the falling-film tube apparatus. It was used successfully with the zinc slurry reagents as well as with the solutions. All measurements were made at room temperature, nominally 20°C and always in the range 19 to 22°C; there was little indication that the liquid-phase resistances varied much with temperature.

**Results with once-run liquors:** There was no indication that liquid-phase resistance by the better reagents varied with the concentration of  $H_2S$  at levels up to 500 p.p.m.; such variation would be expected where the absorption process was slow in the liquid phase. From the results it was shown that good reagents had values of  $BkI$  better than 200 to 350 units, equivalent to values of the gas-phase coefficient  $kg$  in practical board-packed towers for washing fuel gases. It was also shown that aqueous solutions of zinc sulphate varied with acidity. Those with pH 5 or less were quite ineffective.

**Comparative performances of large and small washers:** Board-packed towers are the correct choice for gas-washing processes involving only gas-phase resistance and the highest convenient gas velocity should be used to minimise this resistance. Board-packing is an economical means of achieving the large area of interface necessitated by the type of liquid-phase resistance expected in final purification.

### Packing for Washers

Standard board packing selected for use in both large pilot-plant washers and the proposed full-scale plant is as follows:

Boards—pitch pine,  $3 \times \frac{1}{2}$  in., not serrated.  
Grids—boards at  $\frac{1}{2}$  in. spacing,  $\frac{1}{2}$  in. pitch.  
Arrangement—each grid turned 90° relative to that below. No spacers between the grids.  
Gas velocity—7 ft./sec. between the boards.

Where the falling-film tube is not big enough, small board-packed washers have been used and the standard features of these were as follows:

Tower—Perspex tube,  $\frac{5}{8}$  in. bore, 0.165 sq. ft. in cross section.  
Boards—teak,  $2 \times \frac{1}{2}$  in., not serrated.  
Grids—boards at  $\frac{1}{2}$  in. spacing,  $\frac{1}{2}$  in. pitch, dowelled.  
Arrangement—each grid turned 90° relative to that below. No spacers between the grids.  
Free cross-section—0.096 sq. ft.  
Wetted perimeter—5.67 ft.  
Gas flow—600-1,000 cu. ft./hr.  
Gas velocity—1.74-2.90 ft./sec. between the boards.  
Liquor flow—60 to 100 gall./hr.

Washer covers, liquor distribution troughs and the packing supports were of Perspex and most of the ancillary equip-

ment was constructed from plastics. There was no contamination of the liquor by iron compounds, making liquor analysis much simpler.

As the small board-packed tower was for the study of liquid reagent, it was operated at low gas velocities, well within the viscous range for continuous ducts of width equal to the board spacing. Performance improved slightly with increasing gas throughput, probably due to increasing turbulence caused by crossing of the grids. This showed the dangers of designing washers for use at moderate gas velocities and the need to approach the design by the transfer-unit method rather than in terms of  $kg$ .

It was not practicable to use once-run caustic soda plus zincate for the large-scale calibrations because on recirculation the zincate came out of solution. By combining recirculation of the liquor with a continuous make-up of fresh reagent and an equivalent rejection of used reagent, fairly satisfactory performance was achieved under conditions where  $CO_2$  removal was about  $\frac{1}{2}$  per pass of gas through the tower and the pH value was stable about 11. Measurement of  $H_2S$  concentration with lead acetate papers gave very consistent results. Slip of  $H_2S$  increased steadily with time, giving a satisfactory plot that could be extrapolated to zero time.

### Comparative Testing

Further comparative testing with practical reagents for continuous recirculation will be carried out to determine the effective wetted area of the board-packed towers in comparison with the falling-film tube, and to ascertain the extent to which liquid-phase resistance remains low when liquor is continuously recirculated. Preliminary tests with the solution containing glycine gave a negligible, but unrepresentative, slip of  $H_2S$  because of foaming. This characteristic difficulty with new pitch-pine packings did not occur with the stronger alkali.

**Design of a full-scale washer:** For concentrations of  $H_2S$  up to 5 p.p.m. only a single-stage washer is economically justifiable when operating with slurry reagents for final purification; with the fully dissolved reagent containing glycine, only a single washing stage is necessary even with high purification loads.

The design proposed is for the treatment of 10 million cu. ft. per day of coal gas containing 2%  $CO_2$  and 1 grain HCN per 100 cu. ft., together with the small slip of  $H_2S$  present at the outlet of oxide purifiers operated at a throughput substantially above normal. The  $H_2S$  has to be reduced forty-fold, requiring 3.7 transfer units. Concentration of  $H_2S$  would have to exceed 4 p.p.m. at the washer inlet before the slip exceeded 0.1 p.p.m.

The washer is a board-packed tower with the same packings as the 18-in. bore pilot-plant washers. Assuming that 7/12 of the cross-section is free for gas flow, total cross-section required at 7 ft./sec. between the boards is 28.4 sq. ft., i.e. 5 ft. 4 in. square or 6 ft. diameter.

Irrigation at 5,660 gall./hr. gives 64 gall.=1 cu. ft. of liquor/hr./ft. of wetted

perimeter. Distribution of slurries may be effected with least risk of local accumulation of solids, by parallel pipes each drilled with a single row of small holes to spread the liquor. These distributors cause some splash and a knitted-mesh demister may be provided to conserve liquor containing an expensive organic additive. Gas flow is upwards although the reagent is theoretically suitable for concurrent flow.

**Operation with zinc oxide, glycine and sodium carbonate solution:** The ancillary plant is for removal of zinc sulphide from the washer, recovery of the bulk of the solution by settlement of the zinc sulphide, and of most of the remaining dissolved components by washing the zinc sulphide before its rejection.

Final filters are cheap and simple tanks arranged for gravity drainage. At the standard gas rate, annual accumulation of zinc sulphide is 925 lb. dry weight per 1 p.p.m. of  $H_2S$  removed. Using this system it is hoped to maintain high-activity reagent in the washer, to achieve nearly 100% utilisation of zinc and to conserve the valuable soluble components of the liquor at the cost of an additional steam consumption of about 40 lb./hr.

### Advantages in Chemical Costs

It is possible that the advantages, in chemical costs, of using glycine may barely pay for the extra steam, so that justification for the use of glycine may rest mainly on other advantages of the reagent solution.

**Chemicals consumption and costs:**  
Prices are as follows:

	Price per Ton, £	Price per ton of zinc, £
Zinc acetate dihydrate ..	200	688
Zinc sulphate heptahydrate ..	33	145
Zinc hydroxide ('112%') ..	71-10s	97
Zinc oxide ('97%') ..	71-10s	92
Sodium carbonate ..	15	
Glycine ..	1,080	

Comparative annual consumptions and costs for the standard case, which may be multiplied by load factors for gas quantity and quality, are as follows:

- 1. Zinc sulphate—soda:** Original slurry reagent made from 1 cwt.  $ZnSO_4 \cdot 7H_2O$  + 2 cwt.  $Na_2CO_3$  in 1,000 gall.  
Zinc sulphate 4,540 lb. costing £66.8  
Sodium carbonate 3,020 lb. costing £20.2  
Total cost £87.0
- 2. Zinc hydroxide—soda:** Improved slurry reagent made from  $\frac{1}{2}$  cwt.  $Zn(OH)_2$  + 2 cwt.  $Na_2CO_3$  in 1,000 gall.  
Zinc hydroxide 1,400 lb. costing £44.8  
Sodium carbonate 833 lb. costing £5.6  
Total cost £50.4
- 3. Zinc oxide—soda—glycine:** Improved reagent solution made from  $\frac{1}{2}$  cwt.  $ZnO$  + 2 cwt.  $Na_2CO_3$  + 1 cwt. glycine in 1,000 gall.  
Zinc oxide 772 lb. costing £24.6  
Soda used 104 lb. costing £0.7  
Glycine 52 lb. costing £2.5  
Total cost £27.8

Even 90% recovery of glycine from the zinc sulphide might be considered enough; total cost would then be £40.6.

All other charges for the final purification of 10 million cu. ft. of coal gas/day might add up to £4,000 per annum, equivalent to 0.53d./therm. Chemical costs are small by comparison and are really only important when loads greater than 1 p.p.m. have to be considered.

It may be supposed that the throughput of a set of oxide purifiers could be increased by 33% with equal safety margin if a washer for final purification were made available. This would require an increase in capital investment of only 10% and the overall cost per therm would thus be less.

**Final purification with iron oxide:** Value of final purification by washing, particularly when iron oxide purifiers are included, must be judged in comparison with conventional oxide catch purifiers for the same duty.

In catch boxes, the oxide should be uniformly alkaline and free from liquid water but in contact with gas almost saturated with water vapour, so that it contains about 10% of water physically absorbed like benzene on active carbon. Very little sulphur can accumulate on oxide used in this way because there is no liquid water to act as a medium for the migration of the iron to the outside of the sulphur,

and because slightly acid conditions are needed for this migration.

In the main purifiers, liquid water should always be present in all parts of the oxide to ensure migration of the iron and to act as an evaporative coolant to control heat of reaction and to prevent loss of activity due to drying out.

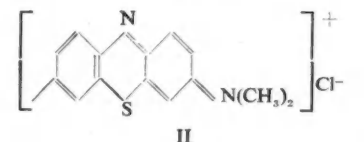
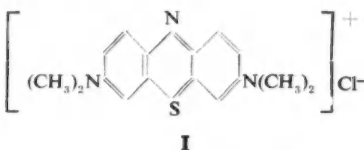
For a fair comparison with the washing methods, catch purifiers should, therefore, contain alkaline oxide of high iron content and high surface area. They should be applied where the gas is saturated with water vapour, but the gas temperature should be kept above the dew-point by means of a small indirect heater.

Work is continuing on the chemistry of alkaline zinc solutions containing glycine, on the comparison of small and large washers using reagents offering some resistance to the absorption of  $H_2S$  in the liquid phase, and on development of proposals for full-scale operation.

## Chemistry of the Methylene Blue Process for Hydrogen Sulphide Removal

IN an appendix to their paper on 'Removal of hydrogen sulphide from gas using an organic oxygen carrier' (see CHEMICAL AGE, 29 November, p. 908), J. E. Davis, J. E. Mills, and C. Ryder, South Eastern Gas Board, describe the chemistry of the methylene blue process for hydrogen sulphide.

Methylene blue, a member of the thiazine group of dyestuffs, is thought to be an equilibrium mixture of the possible ortho and para quinoid structures, I and II.



It is generally obtained as the chloride. Hydrogen sulphide and a wide range of other reducing agents, reduce methylene blue to the colourless leuco compound, which is readily re-oxidised to the original dye. Products of the reduction reaction are the leuco form of the dye and sulphur, and re-oxidation yields the oxidised dyestuff plus water. The exact mechanism of their formation is uncertain.

Experimental work has confirmed that organic bases, e.g. the ethanalamines, will greatly accelerate reduction, but the exact function of the catalyst is unknown. During an experiment using triethanolamine as the catalyst in aniline

solution of methylene blue, a quantity of triethanolamine hydrochloride was isolated and identified and led to the following overall reaction being postulated:

Methylene blue chloride + triethanolamine = leuco methylene blue base + triethanolamine hydrochloride.

Water required for the reaction is present in considerable excess, in the solid methylene blue chloride.

Davis *et al.* concluded that the performance of the solution to be used in their proposed process for removal of  $H_2S$ , would be greatly enhanced if preliminary treatment for chloride were to be carried out. It was found that a simple inorganic base such as ammonium hydroxide was capable of removing the chloride in the same manner as the triethanolamine. The ammonium chloride formed is removed in the aqueous layer while the solution is in the reduced state.

Experiments have been carried out on the velocity of the reduction of the methylene blue base (using aniline as solvent), by  $H_2S$  and the value of the velocity constant  $k$  was compared with values calculated from similar experiments using methylene blue chloride with and without addition of triethanolamine. These have indicated that the catalytic action of triethanolamine involved more than the mere removal of the chloride, but use of the base had a distinct advantage over use of the chloride alone.

Reduction of methylene base was found to be considerably slower in solvents other than aniline, which were either neutral or acidic. Davis *et al.* therefore conclude that the primary function of the basic catalysts was to provide a strongly basic medium and that promoted an accelerated reduction of methylene blue, either as chloride or base.

## Gas Research Meeting

## Commercial News

### Boots' Pure Drug

A new profit earning scheme under which 8½% of the profits will be set aside each year as a bonus fund for employees has been announced by Boots Pure Drug Co. Ltd. Distribution of the bonus to the company's 40,000 employees will be based on salary and length of service.

The scheme replaces a prosperity bonus for which funds have been set aside in previous years at the discretion of the directors. In the last financial year this bonus amounted to approximately 7½% of profits. This cost the company a total of £415,000. On the basis of last year's profits the new 8½% bonus would amount to £476,000.

Chairman of the company, Mr. J. P. Savage, in an explanatory leaflet, states "If the profits go up bonus will go up. If the profits go down, bonus will go down. The amount of bonus each member of the staff will receive will depend on the profits earned."

### British Benzol

In the year ended 31 October, British Benzol and Coal Distillation incurred a net loss of £28,976 (profit of £22,330). The dividend is being cut from 15% to 10% with a 5% final.

The loss is after crediting £2,069 tax recoverable (£47,600 paid). With a non-recurring trade dividend of £20,604 and £57,997 (£67,230) brought in, there is £49,625 available against £89,560. Expenditure of £35,276 incurred on essential services during the year has been charged against future depreciation reserve.

### Gas Purification

Results of Gas Purification and Chemical Ltd. for the first four months of the current year have shown a profit of £75,712, the new chairman, Vice-Admiral Sir Charles Hughes Hallett, reports. This figure is unaudited and includes the holiday months.

### McKechie Bros.

Group profit of McKechie Bros., after meeting all charges except tax, was £527,609 (£393,823) in the year to 31 July last. This increase was due mainly to improved trading by the parent company.

Capital additions during the year amounted to £833,192. Expenditure by the parent company totalled £510,254, of which £415,983 was spent on the new factory at Aldridge. Subsidiary companies' expenditure totalled £322,938, of which £294,304 was spent overseas. Capital commitments total £442,000.

Final dividend of 10% has been recommended making 15% (same).

While trading on the metals side had improved, a serious setback had been experienced on the chemicals side, the company's production of sulphate of

- 8½% of Boots' Profits for Staff Bonus
- British Benzol Cut Dividend for Year
- McKechie's Concern at Copper Sulphate
- U.S. Croda Acquire Lanolin Division

copper being at its lowest for about 30 years. Demand for sulphate of copper was exceptionally poor, due mainly to a carry forward of stocks in the hands of manufacturers, merchants and consumers. This situation had led to intensive competition for orders.

The fall in the price of copper had caused stocks to suffer a further depreciation of £127,000 (charged against revenue).

### Griffiths Hughes

Half-yearly dividend on the 5½% cumulative preference stock of Griffiths Hughes Proprietaries Ltd. will be paid on 31 December, 1958. The directors have declared an interim dividend on the ordinary stock of 2½%, less tax, on account of the year ending 31 March.

### United Indigo and Chemical

Payment of the preference dividend of United Indigo and Chemical Co. Ltd., which is due on 31 December, is being deferred until the accounts for the full year to 30 June 1959 are available.

### Anglo-Lautaro Nitrate

Dividend of \$(U.S.)1 per share on class 'A' shares for the year to 30 June 1958 and corresponding pro rata amounts on 'B' and 'C' shares, are announced by Anglo-Lautaro Nitrate Corporation. No dividends were paid for 1956-57.

## Market Reports

### COPPER SULPHATE WELL HELD AT HIGHER PRICE

**LONDON** Home demand for industrial chemicals has been on a fair scale with a steady flow of contract delivery specifications. The plastics industry continues to be a good outlet and there is a steady call from the paint trade.

Prices generally are unchanged and firm with dearer quotations reported for the chemical compounds of the non-ferrous metals. Latest reported price for zinc oxide, red seal, is £95 10s. per ton. Sulphate of copper is well held at the higher level and elsewhere a good call is reported for hydrogen peroxide and formaldehyde.

Among the coal tar products, pitch is in good request on home and export account and there is a fair demand for refined tar, creosote oil and cresylic acid.

**MANCHESTER** The price position has been maintained in virtually all sections of the Manchester chemical market during the past week. The textile

and allied industries are taking reduced quantities, but contract deliveries to most other industrial outlets have been fairly well maintained. Replacement business has been moderate. Exports continue on reasonably steady lines. Basic slag and the compounds are still the most active sections of fertilisers.

**GLASGOW** Quite a good level of business was maintained during the past week on the Scottish heavy chemical market. Demands against contract requirements were fairly well taken up; spot demands were made for a varied range of chemicals. Some interest is also being shown in regard to contract renewals for next year. On the whole prices have continued firm.

### Croda Inc., U.S.

U.S. subsidiary of Croda U.K., Croda Inc., have acquired the issued capital of the Hummel Lanolin Corporation, which was recently formed to take over the lanolin and wool grease division of the Hummel Chemical Co. Inc. Hummel Lanolin will be a subsidiary of Croda Inc.

It is also announced that Croda Inc. have established a lanolin manufacturing plant at Newark, New Jersey. This plant will supply the Croda organisation in the U.S., with lanolin and its derivatives.

### INCREASES OF CAPITAL

**BLAW KNOX CHEMICAL ENGINEERING CO. LTD.** Increased by £199,900 beyond the registered capital of £100.

**MILTON PHARMACEUTICALS LTD.** (formerly Milton Industrial Chemicals (London) Ltd.), 10 New Burlington Street W.1. Increased by £369,900 beyond the registered capital of £100.

The export market still shows considerable interest with a volume of inquiries being received. The position regarding agricultural chemicals still emphasises forward requirements.



## Overseas News

### EAST GERMANY TO RAISE SULPHURIC ACID CAPACITY TO 1 MILLION TONS

DETAILS have been released in the Federal Republic of a further stage in the chemical production programme of the East German Republic, a country which is at present concentrating much energy on an expansion of its already comparatively important chemical industry. The industry is scheduled to account ultimately for one-third of total national production.

First step in the new plan is the raising of the country's sulphuric acid capacity to 1 million metric tons by 1970. A sulphuric acid plant has been built at Salzwedel at the cost of 10 million East Marks (cross-rates West to East equal 4:1, 10 million West Marks equal approx. £830,000) and with an initial daily production of 60 metric tons; it is to come into production in the next few weeks. The erection of a second plant on the same site and with double the capacity of this is to be undertaken under the country's third five-year plan within the next few years. A sulphuric acid plant at Coswig is to be finished soon, too, and will start production with an annual 60,000 metric tons; this plant is due to produce 220,000 metric tons of sulphuric acid a year by 1963.

An artificial fibres plant at Premnitz is to be extended to such an extent as to become the largest works of its kind in Eastern Europe. Some East Marks 200 million (West Marks 200 million equal approximately £16½ million) will be invested in this expansion scheme, 195 million Marks of it (195 million West Marks equal £16½ million) for the production of an acrylic fibre. This acrylic, which has not been made previously at the plant, will be produced in quantities of up to 10,000 metric tons a year.

The Buna synthetic rubber plant near Halle is expected to become the largest producer of carbide in the world, it is claimed. Increased chemical production will also be based on the country's large resources of potash and lignite.

Next March, Russia is due to hand over blueprints for a petrochemical works which will process 5 million tons of Soviet crude oil to be piped to East Germany from Baku, and 1 million tons of oil from domestic wells.

#### New Division of A.W.'s Canadian Subsidiary

Electric Reduction Company of Canada Ltd., Albright and Wilson's Canadian subsidiary, announced last week that a new division, ERCO Engineering Services, has been established to design, construct, install and start up tailor-made chlorine dioxide plants for pulp bleaching or other uses. The division will design plants to fit

into the particular location required and sell on a 'turn-key' basis with a guaranteed minimum operating efficiency. Training of personnel will also be undertaken. It is believed that the new service is unique in the Canadian chemical industry.

Director of the division is Mr. W. R. Cale, who was manager of the research and technical service department of Electric Reduction when early work in the development of chlorine dioxide generation was being carried out.

#### Dow Chemical's Contract for Soviet Benzene

Russia's biggest post-war success in the sale of cut-price raw materials in the U.S. is represented by an agreement by Dow Chemical to purchase \$13.5 (£4.8 million) worth of benzene from the U.S.S.R. This is not the first import of Russian benzene into the U.S. market.

Price of the benzene will be equal to 24-25 cents a gallon, against the current U.S. price of 31 cents. The price of benzene used in the production of phenol and styrene fell by 5 cents during this summer.

It is understood that Dow Chemical's decision has been influenced by serious fluctuations and deficiencies in domestic supply in past years, although these no longer occur.

#### Du Pont in Benelux

E.I. du Pont de Nemours and Co., who recently stated that a subsidiary company is to be set up in Belgium, now announce that another company is to be formed for the production of synthetic fibre and plastics within the Benelux area as a whole. A commission of experts appointed by the company is now deciding where the new subsidiary will be based; present reports indicate that this will be either Dordrecht, near Rotterdam, or Mechelen, Belgium.

#### B.A.S.F. to Raise Capacity for Methanol

Badische Anilin- und Soda-fabrik (B.A.S.F.) have started on extensions to their methanol plant. The extended manufacturing installation will come into operation at the end of this year. At present B.A.S.F., with a monthly methanol production of more than 10,000 m.t., supply half of the total production in West Germany.

#### German Dye Producers set up U.S. Subsidiary

A U.S. subsidiary of the West German chemical concern of Vereinigte Ultramarin Fabriken A.G., Cologne, has been

set up in New York under the name of United Ultramarine and Chemical Co., the Cologne company reports. The main business of this company will be to set up a sales network for the German concern in the U.S. and in Canada. Further, a closer contact with U.S. chemical producers is the aim, with a view to obtaining licences for the manufacture of patented goods and the use of patented processes. An agreement between Vereinigte Ultramarin and American Cyanamid Co., which formerly made ultramarine dyes itself and later handled the products of the German concern, has come to an end.

#### High Purity Silicon via New Iodide Process

Footo Minerals are due to start up a plant on a semi-commercial scale initially to furnish high purity silicon using a new iodide. The plant should reach full production next year. Thermal reduction of silicon tetrachloride purified by distillation will produce silicon pure enough for solar batteries and low power grades (2 to 10 parts per 1,000 million impurities). Subsequent zone refining will lower the impurities to 1 part per 1,000 million.

In the U.S. the market in 1958 for high purity silicon is estimated to amount to about 50,000 lb. The current annual rate is reported to be about twice the above figure.

Of U.S. silicon producers, E. I. Du Pont de Nemours and Co. are the largest, Merck Inc., however, are the only producers of ultrapure (1 part per 1,000 million) silicon on a commercial scale.

#### New Solvay P.V.C. Plant

Recently opened in Rheinberg, West Germany, is the seventh p.v.c. plant of the international Solvay Group. The plant, operated by the Deutsche Solvay-Werke G.m.b.H., has an annual capacity of some 100,000 metric tons.

#### U.S. Sulphur Experts Visit to Poland

Mr. F. Wilson, vice-president of the Freeport Sulphur Co., and Mr. D. Flint, the company's chief geologist, visited Poland recently at the invitation of the Ministry of the Chemical Industry. Their trip included visits to the Tarnobrzeg sulphur mining area and to the Wieliczka salt mines.

#### Acetic Acid for Acetate Solvents

Since acetic acid is widely used in the manufacture of acetate solvents for the preparation of nitro cellulose lacquers, National Chemical Products Ltd., Transvaal, have constructed and put into operation a new acetic acid plant which makes them independent of outside supplies.

The plant, in stainless steel throughout, was fabricated and erected by the company's own engineering staff, and it is interesting that the starting material is ethyl alcohol which is one of the company's primary fermentation products.

The alcohol is vaporised, mixed with air, and passed over a catalyst of granular silver, which effects oxidation to crude acetaldehyde. This is purified by fractionation which involves complications because of the reduced atmospheric pressure in Johannesburg and district, where the acetaldehyde boils at only 15°C. To raise this to normal b.p., 20.8°C, pressurised equipment has to be used. In the oxidation to acetic acid, manganese acetate is used as catalyst and final purification is by fractional distillation.

### Uranium Production In Italy

From surveys already made, Italian experts now reckon that in three years Italy will be able to produce 190 tons of uranium annually. Of this total, 70 per cent will come from Val Gardena and other parts of the Eastern Alps and the remainder from Permian shales of the Alpi Cozie and Alpi Maritime. Predicted output is likely to increase as promising minerals have been found in other parts of the country.

### Bacterial Activity in JP-4 Fuel

Certain types of bacteria thrive in kerosene-type jet aircraft fuel and form sludge which could easily affect filter systems. This finding resulted from studies of sludge deposits causing malfunctions of refueling equipment in KC97 aircraft and of fuel systems in B47 aircraft, hampering flight operations of some USAF tactical units. It was indicated that certain types of bacteria find nutritive material in JP-4 fuel. The bacteria emulsify the fuel to form gummy residues, then themselves form precipitates. It was further demonstrated that a 1.5-2 per cent concentration of sodium tetraborate, by weight, or a 2 per cent concentration of potassium tetraborate, by weight, in bottoms of JP-4 storage tanks can produce bacteriostatic conditions. Details of evaluations of the corrosion inhibitors and gum inhibitors are presented in a report by S. Bakanauskas, Wright Air Development Centre, US Air Force (Order PB131813 from OTS, US Department of Commerce, Washington 25, D.C., 75 cents), along with descriptions of methods and results.

### Japanese Plant for New Brazilian Fertiliser Factory

A Japanese company, Toya Koatsu, of Tokyo, are to supply plant for the erection of a new nitrogen fertiliser factory by Brazilian company Nitrogênio S.A. at Salvador, in Bahia State. Capital of Nitrogênio is held by Bracapa S.A., São Paulo, and Chemische Holding A.G. and Chemical Credit and Trading Corporation, both of Zurich.

### W. German Plastics Output Rise is Down by 50%

For the whole of 1958 the plastics materials produced in West Germany will total an estimated 630,000 metric

tons, against 561,000 metric tons last year. The rate of growth has, however, slackened off by nearly 50%; production during the first three-quarters of the year was, at 469,000 metric tons, 13.5% higher than the total for the same period of 1957, while this total was in its turn about 25% higher than the 1956 period figure.

With constant investments, the industry now accounts for 11% of the country's total turnover in chemical products. In 1955 this share was 9%; in the period from the start of that year until the end of 1957 some DM550-600 million (£47-£50 million) has been spent on the plastic industry's development and enlargement, some DM220 of this (about £19 million) in 1957 alone.

Latest figures show that polymerisation products now account for 45% of all plastics made in West Germany, and in quantity produced have risen in the past year by 16%; condensation products form 41% of the total and show a quantity rise of 13% over the year; the cellulose derivatives are up by only 8% on 1957.

### Indonesia Nationalises Large Dutch Chemical Firm

Indonesia's leading chemicals and pharmaceuticals concern, the Dutch N.V. Chemialienhandel Rathkamp, has been taken over to be operated as a nationalised concern by the Indonesian Government.

### New Hungarian Fertiliser Plant Boosts Production

Hungary's latest artificial fertiliser factory, the Borsod Integrated Chemical

Plant reached its expected annual production of 40,000 tons in November.

By the end of the year output should reach 50,000 tons, giving the country almost 50% more nitrogenous fertiliser than was previously available. The plant started operating in May and full production was attained in September.

### China's New Chemical Centre

The new chemical manufacturing centre for Communist China, which is to be built at Kirin, in Manchuria, will include plants for the production of synthetic fibres, plastics and synthetic rubber. The country's first plastics plant, in Peking, at present under construction, will produce phthalic anhydride, p.v.c. and soda.

### Olin Mathieson's Marketing Headquarters for Organics

Chemicals Division of Olin Mathieson Chemical Corporation, New York, US, have consolidated their national marketing headquarters for organic chemicals at 745 Fifth Avenue, New York. The move has been made to speed customer service.

### Bayer-U.S. Rubber Agreement

Farbenfabriken Bayer A.G., Leverkusen, and the Naugatuck Chemical Division of the United States Rubber Co. have signed an agreement under which they will exchange technical advice and patent licences for the manufacture of products for the rubber industry.

## Ziegler's Work on Synthetic Rubber

DETAILS of research carried out at the Max Planck Institute for Coal Research, Mulheim-on-Ruhr, on the use of metallic alkyls in the production of a new type of synthetic rubber were given recently by Professor Karl Ziegler. He was speaking at a conference of the German Society for Mineral Oil and Coal Chemistry at Goslar.

Organo-metallic compounds, particularly aluminium compounds, were used as a basis for the experiments, said Dr. Ziegler, the research being initially concerned with the so-called immediate synthesis of aluminium trialkyl from aluminium, hydrogen and olefins and with the various uses to which such a product could be put; that was as catalysts and as true intermediate products. The alkyls could be used either for catalysing purposes or as auxiliary agents in the production of extra-active organo-metallic co-catalysts as was known. With the aluminium alkyls above, olefins, particularly ethylene, in the low-molecular field could be polymerised. This meant that using ethylene, butylene, hexene, octene etc. or with propylene or isohexene, a most varied range of

materials could be produced.

By the addition to such chemicals as listed above of the aluminium alkyls and titanium compounds it was possible to effect a favourable physical development into the high-molecular range, which made possible the production of any number of quite different types of product. Reaction with catalysts such as the above yielded products such as low-pressure polythene, polypropylene, various types of butadiene-rubber and so on. A catalyst agent of this type had already, outside Mulheim, been able to produce what was called 'synthetic natural-rubber' from isoprene. Another organo-metallic co-catalyst had been found to convert butadiene under cyclic trimerisation into cyclododecatene, a basis for many interesting new compounds, particularly in the field of Perlon-type materials.

The intermediate products were the organo-aluminium compounds used for the production of higher-quality fatty alcohols from ethylene, organic borax compounds and of tetra-ethyl lead (the best-known anti-knock medium for motor engines).

● **MR. P. W. LUCAS**, who has been appointed sales office manager of the Chemicals Division of Union Carbide Ltd., was formerly a senior purchasing officer with Bakelite, in which capacity he was primarily responsible for the purchasing of imported raw materials. **MR. A. P. MARSDEN** has been appointed traffic manager of Union Carbide. This department has been formed to deal with the centralisation of the company's Alloys and Chemicals Divisions and its present expansion plans.

● **MR. F. C. HYMAS**, chief chemist, Spratt's Patent Ltd., was elected chairman at the recent annual meeting of the London section, Royal Institute of Chemistry, to succeed **DR. C. C. HALL**, senior principal scientific officer, at the D.S.I.R., Warren Spring Research Station, Stevenage. Other officers elected were: vice-chairman, **P. A. RAINE**, chief chemist, Johnson and Phillips Ltd., Charlton; hon. treasurer, **P. F. CORBETT**, Shell-Mex and B.P. Ltd. The following remain in office: vice-chairman, **DR. J. H. PRYOR**, assistant professor of chemistry, Royal Naval College, Greenwich; hon. secretary, **DR. S. A. MILLER**, basic research manager, British Oxygen Research and Development Ltd., London S.W.19; hon. assistant secretary, **G. C. ACKROYD**, principal scientific officer, D.S.I.R. Fuel Research Station, Greenwich. Elected to the committee were: **R. J. COLE**, **T. DEWING**, **W. C. A. HARDS**, **F. L. HULBERT**, **G. H. JEFFREY**, **N. LINDOP** and **C. SIMONS**.



R. W. Rutherford



F. C. Hymas

● **MR. R. W. RUTHERFORD**, the chairman of the new Nuclear Chemical Plant Ltd., 22 Carlisle Place, London S.W.1, joined the board of the Power-Gas Corporation, Stockton-on-Tees, in 1948 and became deputy managing director in 1953. He is also a director of the associated company, Rose, Downs and Thompson Ltd., Hull. Mr. Rutherford is a member of the Institution of Chemical Engineers, the Institute of Gas Engineers and the Institute of Fuel. In promotion of the firm's business he resided in Japan for a number of years and since his return to the U.K. has travelled widely abroad.

● **MR. R. A. C. ISBEL**, A.Inst.P., technical sales manager, Hilger and Watts Ltd., 98 St. Pancras Way, London N.W.1, was re-elected chairman of the Physical Methods Group, Society of Analytical Chemistry, at the recent annual meeting. **MR. G. W. C. MILNER**, M.Sc., F.R.I.C., A.Inst.P., senior principal scientist, Atomic Energy Research Establishment, Harwell, was re-elected

## PEOPLE in the news

vice-chairman, and **DR. T. L. PARKINSON**, B.Sc., F.R.I.C., Production Research Division, Béchams Foods Ltd., Great West Road, Brentford, was elected hon. secretary and treasurer in succession to **MR. L. BREALEY**, Boots Pure Drug Co. Ltd.

● **MR. D. VAN ZWANENBERG** has been appointed manager of Allied Distributing Corporation Ltd., sole distributors of Jenolite domestic products, etc., and Jenograph Ltd., makers of natural graphite lubricants. Mr. Zwanenberg was formerly area manager of Deosan Ltd.

● **MR. HELLMUTH CARROUX**, former president of the Association of German Wholesalers, Importers and Exporters of Chemicals and Drugs, has been appointed president of the association. He will represent the German chemical trading industry at all international meetings and top-level conferences. His post as president has been taken over by the former vice-president **MR. EBERHARD LEINS** (Herkomer and Bangert of Stuttgart).

● **MR. L. H. ADCOCK**, B.Sc., F.R.I.C., an experienced analyst who for the past 20 years has been with Howards of Ilford Ltd., has joined the staff of the Printing, Packaging and Allied Trades Research Association. Mr. Adcock, who headed the company's research analytical group, has also had experience in the use of radioactive tracer techniques for metabolic studies. He has contributed articles on these studies and related analytical work to *The Analyst*.

● Board of the newly formed Birlec-Efco (Melting) Ltd., Westgate, Aldridge, Staffs, comprises: **GEORGE P. TINKER**, chairman; **J. C. HOWARD**, managing director; **F. S. LEIGH**, assistant managing director; **D. L. CAMPBELL**, **J. H. CROSSLEY**, **P. F. HANCOCK**, **J. A. MONKS** and **T. G. TANNER**. Secretary is **J. C. MANTELL**.

● **MR. W. K. MCGAVIN**, M.Sc., who has succeeded **MR. F. MACKLEY** as manufacturing director of Shell Chemical Co. Ltd., joined Shell of New Zealand in 1933 as assistant chemist. He came to the U.K. in 1934 and after holding various posts at home and abroad he became superintendent of the Stanlow chemical plants in 1952. His last post abroad was

that of refinery manager at Suez which ended abruptly with the Suez incident in 1956. Other Shell Chemical appointments include **DR. J. L. EDGAR** as manager, manufacturing operations department, **MR. J. O. DUGDALE BRADLEY** as manager, engineering department and **MR. L. HOLLIDAY** as manager, technological department. Chemical plant appointments include **MR. R. W. LERRIGO** as manager at Stanlow and **MR. E. J. DAWSON** as manager at Shell Haven. At the Partington Works, **MR. W. E. HUGGETT**, general manager, will be assisted by an administrative manager, **MR. F. W. A. PATERSON** and an operations manager, **MR. H. L. STURGESS**.

● **PROFESSOR KARL ZIEGLER** of the Max Planck Institute for Coal Research, Mulheim-on-Ruhr, was recently presented with the Carl Engler Medal, the highest award of the German Society for Mineral Oil and Coal Chemistry. The award was made for his work in the use of metallic alkyls in the production of a new type of synthetic rubber and for other research connected with hydrocarbon chemistry.

● **MR. RICHARD J. WINGROVE**, Birlec Ltd., has been awarded the William Macnab Medal of the Institution of Chemical Engineers for



R. J. Wingrove

1957, presented annually for the best set of answers in the associate membership examination. Mr. Wingrove, who obtained his M.Sc. at Reading University for research in chemistry, was a part time student at the Battersea Polytechnic from 1954 until he completed the institution examination in 1957. He started his career as an assistant chemical engineer with Head Wrightson Processes Ltd. and then spent several years as a chemist in the Fulham Research Laboratory of the North Thames Gas Board. In July 1958 he joined the Drier and Gas Plant Division of Birlec Ltd., Birmingham 24, as a project engineer and has since been working on the design of gas generators.

● **MR. ROBERT S. FIRST**, industrial consultant, 6 East 39th Street, New York 16, N.Y., U.S., has been retained by 12 U.S. chemical companies to visit chemical and plastics companies in England, France, Switzerland, Sweden, West Germany, Holland, Belgium and Italy. The objectives of this work will be to determine what processes and products in Europe in the chemical and plastics fields are available for licence in the U.S. Mr. First is now in London and will be in London again early in January.

● **SIR ERIC RIDEAL**, M.B.E., F.R.I.C., F.R.S., elected chairman of the new Surface Activity Group, Society of Chemical Industry, was formerly a director of Monsanto Chemicals Ltd. He is also a former professor of chemistry of King's College, London University. Hon. secretary of the new group is **MR. M. K.**



SCHWITZER, Armour Chemical Industries Ltd., 4 Chiswell Street, London E.C.1, and the hon. recorder is MR. F. RILEY, Marchon Products Ltd., 140 Park Lane, London W.1.

● MR. H. N. WIGAN, M.C., M.Inst.Pet., who recently resigned from the board of the Amber Chemical Co. Ltd., has joined the board of Combustion Chemicals Ltd., 33 Dorset Square, London, N.W.1. This company now holds the licence for the manufacture and sale throughout the U.K. and Commonwealth of the well-known French fuel additives and sulphur inhibitors, Desulfur, Dieselfur and allied products.

● DR. J. G. PEARCE, C.B.E., director of the British Cast Iron Research Association, retires at the end of 1958 from the post he has held since 1926. He will be succeeded by MR. H. MORROGH, the deputy director. The period 1924-1958 has been one of marked growth and change; B.C.I.R.A., which in 1924 had a staff of about 12 and had an income of about £5,000, now has a staff of 150 and an income provided by industry and the D.S.I.R. of about £200,000. A dinner to mark his retirement will be given to Dr. Pearce at the Connaught Rooms, London, on 15 December. DR. H. T. ANGUS, now assistant director, has been appointed deputy director from 1 January; MR. T. E. WHITESIDE will continue as secretary.

● MR. M. H. J. VILLENEUVE, former head of the chemical section of the commercial development department of Pfizer Ltd., Folkestone, has been transferred to Kemball, Bishop and Co. Ltd., as technical service manager. This is a new post for Kemball, Bishop, who were acquired by Pfizer in October.

● At the anniversary meeting on Monday of the Royal Society SIR CYRIL HINSHELWOOD, Dr. Lee's professor of chemistry at Oxford, was re-elected president. Other officers re-elected were: treasurer, SIR WILLIAM PENNEY, director, Atomic Weapons Research Establishment, Aldermaston; biological secretary, SIR LINDOR BROWN, Jodrell professor of physiology, University College, London; physical secretary, PROFESSOR W. V. D.

HODGE, Lowdean professor of astronomy and geometry, Cambridge; foreign secretary, DR. H. G. THORNTON, lately head of the department of soil microbiology, Rothamsted Experimental Station. Other members of Council elected included: SIR CHARLES DODDS, Courtauld professor of biochemistry, London University, PROFESSOR G. GEE, C.B.E., Sir Samuel Hall professor of chemistry, Manchester University, DR. H. GODWIN, reader in quaternary research, Cambridge, PROFESSOR R. D. HAWORTH, Firth professor of chemistry, Sheffield University, PROFESSOR A. L. HODGKIN, Royal Society research professor, PROFESSOR D. M. NEWITT, M.C., Courtauld professor of chemical engineering, Imperial College.

● Owing to ill-health, MR. J. S. HATFIELD has relinquished his position as executive director of Wm. Butler and Co. (Bristol) Ltd., St. Philips, Bristol. His duties will be assumed by MR. W. J. STIMPSON, who was recently appointed general manager. Mr. Hatfield is retaining his seat on the board.

● Three prominent chemists are among four U.K. scientists who will make lecture visits to Russia starting next spring. The four are: PROFESSOR GEOFFREY GEE, Sir Samuel Hall professor of chemistry, Manchester University; DR. F. P. BOWDEN, reader of physical chemistry, Cambridge University; PROFESSOR C. E. H. BAWN, Grant-Brunner professor of inorganic and physical chemistry, Liverpool University; and PROFESSOR E. N. DA C. ANDRADE, emeritus professor of physics, London University.

● MR. J. FRISKEN has been elected vice-president of the Fertiliser Society in succession to MR. A. I. COLEMAN, who has temporarily reduced his commitments owing to his recent ill-health.

#### U.K. Firm Supplies World's Largest Flasks for Russia

Seven 200-l glass flasks, the world's largest, are among equipment being supplied by Q.V.F. Ltd., for a Russian factory which will manufacture cellulose acetate yarn. The flasks are being supplied to Courtaulds, who are supplying machinery and equipment.

#### US Sales Tour for Pye's Argon Chromatograph

After a successful tour in West Germany, resulting in several large orders, the Pye argon chromatograph is now being demonstrated throughout the U.S. Mr. R. S. Evans, of W. G. Pye



and Co. Ltd., in conjunction with their distributors, the Jarrell Ash Co., has planned a three-month itinerary.

It is anticipated that within the next three months several hundred Argon chromatographs will be installed in laboratories throughout the world.

#### I.C.I. to Stop Making Phenolic Powders

AT OR shortly after the end of the year, Imperial Chemical Industries Ltd. are to stop making phenolic moulding powders. The company will thus be able to concentrate on newer lines and the plant and equipment which will be freed will be used to increase polythene output at Wilton.

While production of phenolic powders in the U.K. is still on a large scale, growth in output has been slowing down for some years, as they are being replaced by the newer thermoplastic materials which are more versatile.

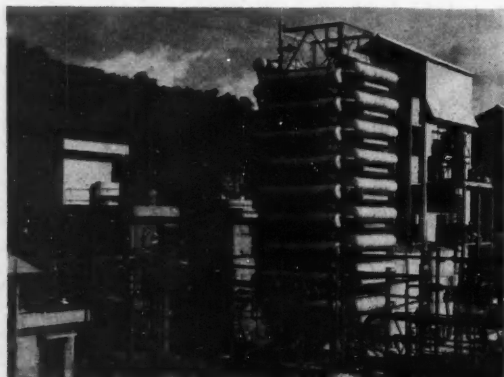
Continuity of supply is assured by James Ferguson and Sons, wholly-owned subsidiary of Wall Paper Manufacturers Ltd., who will take over production and marketing of the same grades of phenolic moulding powders as supplied by I.C.I.

#### T.E.L. Exports Decontrolled

Goods freed from export licensing control from 1 December, under the Export of Goods (Control) (Amendment No. 4) Order—S.I. 1937-1958—include: additives for lubricating oils and greases, chlorinated paraffin wax, certain aircraft fuel blending agents, certain cocks and pipe valves, pipes, metal tubes and pumps partly made of or lined with non-corrosive material, specified nitrogenous fertilisers, glycerol, certain hydrazine salts, nickel hydroxides and oxides, tetraethyl-lead and mixtures.

p-Nitromethylaniline is included among goods placed under export licensing control for the first time.

#### UK Chemico's Nitric Acid Plant at Gomia



This nitric acid plant of 10 tons/day capacity was supplied and recently commissioned by Chemical Construction (G.B.) Ltd. for the new commercial explosives factory of Indian Explosives Ltd. at Gomia. Nitric acid is produced by the pressure oxidation of ammonia

## TRADE NOTES

### New A.P.V. Equipment

Among the new publications of A.P.V. Co. Ltd., Manor Royal, Crawley, Sussex, are three booklets describing special plant. One covers alcohol and yeast equipment and it is stressed that services provided cover the entire contract, i.e., development, design, erection and instrumentation. Another describes batch benzole plants, while the third shows a new plate evaporator.

### Pressure Vessels by Cochran

Illustrations of some of the pressure vessels built by Cochran and Co., Annan, Ltd., Annan, Dumfriesshire, are given in a new booklet just published. These tanks are class 1, fusion-welded vessels for corrosive and non-corrosive fluids.

### New Barytes Source Available

The grinding plant installed by C. Maffei, Milan, in Sardinia, to process Sardinian barytes is now in full production and three standard grades of white barytes ground to 325 mesh are available for immediate delivery from stocks held in England by Guest Industrials Ltd., 81 Gracechurch Street, London E.C.3, and in Scotland by H.M. Roemmele and Co. Ltd., 65 West Regent Street, Glasgow C.2.

### D.D. Lakk

In our reference to D.D. Lakk, the new alkyd-isocyanate polyester resin

available from C. J. W. Macaulay, 60-66 Wardour Street, London W.1 ('Trade Notes,' 29 November) it was incorrectly stated that the product was able to withstand high temperatures of 180-120°C; this should have read '180-200°C.'

### Barium Sulphate Paste

The Board of Trade is considering an application for exemption from import duty of aqueous barium sulphate containing between 70% and 80% by weight of barium sulphate and containing in the dried material not more than 4 p.p.m. by weight of iron compounds, calculated as metallic iron. The material is used for the baryta-coating of paper.

### New Evershed Sales Office

Evershed and Vignoles Ltd. opened, on 1 December, a district office at 14 Yarm Lane, Stockton-on-Tees (Stockton 64288). Their office at 116 Manningham Lane, Bradford 8, will continue to accept overall responsibility for the Northern Counties and requests for service may be made at either office.

### Birlec Drier Division Renamed

In view of the expanding business of Birlec Ltd., Birmingham 24, in adsorption plant and gas generators, the division handling these projects, formerly known as the Drier Division, has been renamed Drier and Gas Plant Division. The new division will continue to be administered by T. C. Sanders, manager,

H. J. Podmore, sales manager, and S. A. Gregory, technical manager. The change in name has been occasioned by the increasing industrial importance of inert gases. Birlec equipment in this field now covers low-humidity air conditioning plant, driers for high pressure applications and specialised gas generators.

### Ashe/Aspro Deal

In an agreement completed between Ashe Laboratories Ltd., of Leatherhead, makers of Amplex deodorant tablets and allied preparations for human and veterinary use, and Aspro-Nicholas Ltd., of Slough, Ashe have acquired the Shirley range of pet foods and medicines from Aspro-Nicholas.

### Change of Address

The Manufacturing Division of Shell Chemical Co. has been moved to the company's offices at Marlborough House, Great Marlborough Street, London W.1.

### De Havilland Adopt 'Gredag'

'Gredag' molybdenum disulphide grease (grade MP 30) has been adopted as a titanium pressing lubricant by De Havilland Aircraft following tests carried out on this Acheson product by their production research department. Referring to the cost of the lubricants, De Havilland's stated in their report that grade MP 30 costs one-third that of a comparable product tested, and was shown to be the most efficient lubricant for the production of hot titanium pressings.

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# NEW PATENTS

By permission of the Controller, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sale Branch), 25 Southampton Buildings, Chancery Lane, London WC2, price 3s 3d including postage; annual subscription £8 2s.

Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

## AMENDED SPECIFICATION

On sale 7 January 1959

Polybasic aromatic carboxylic acids. Henkel & Cie, Ges. 733 322

## ACCEPTANCES

Open to public inspection 31 December

Cellulose triacetate textile materials. British Celanese Ltd. 807 192  
Process and apparatus for purification of crystals. Phillips Petroleum Co. 807 100  
Isomers of pseudoirones substituted at C9 and/or C11. Givaudan & Cie. S.A., L. 807 101  
Underground gasification of coal. Coal Industry (Patents), Ltd. [Addition to 762 484.] 807 195  
Antioxidant compositions. American Cyanamid Co. 807 102  
Methods and devices for testing for the presence of water in suspension in other liquids. Shell Research, Ltd. 806 928  
Apparatus and method for determining hydrogen content in hydrocarbon substance. Standard Oil Co. 807 103  
Method of concentrating latex by evaporation. Triggs, W. W. (Goodyear Tire & Rubber Co.). 806 877  
Polyarylenethylenes and their copolymers. Du Pont de Nemours & Co., E. I. 807 196  
Purification of alkali metal salts of phenoxy-ethylpenicillin. Imperial Chemical Industries, Ltd. 806 929  
Rotary positive displacement device for liquids. Liquid Controls Corp. [Addition to 754 984.] 807 197  
Polymeric materials. Du Pont de Nemours & Co., E. I. 807 198  
Production of 5-(4-hydroxybutyl) hydantoin and intermediates therefor. Du Pont de Nemours & Co., E. I. 806 931  
Amides and process for the manufacture thereof. Roche Products, Ltd. 806 879  
Coating composition. Imperial Chemical Industries, Ltd. 807 199  
Mercurial diuretics and preparation thereof. Sterling Drug, Inc. 806 880  
Method of dehydrating liquid hydrocarbons. Vereinigte Glanzstoff-Fabriken A.G. 807 200  
Rubbery composition and article made therefrom. Goodrich Co., B. F. 806 933  
Presentation and display of heat-exchanger gas activity information in graphite moderated power reactors. Sunvic controls, Ltd. 807 048  
Production of steroid compounds. Laboratoires Francais de Chimiotherapie. 807 203  
Fertilisers. Fisons, Ltd. [Cognate application [7 311].] 807 107

Process for polymerising olefins. Hercules Powder Co. 807 204  
Polycondensation of diglycol terephthalate. Vereinigte Glanzstoff-Fabriken A.G. [Addition to 720 120.] 806 934  
Production of aliphatic nitriles. Lonza Electric & Chemical Works, Ltd. 806 902  
Manufacture of organic isocyanates. Imperial Chemical Industries, Ltd. 806 903  
Reacting carbohydrates and polyhydric alcohols derived therefrom with various reagents. Fairweather, H. G. C. (General Aniline & Film Corp.). 806 935  
Modified polyvinyl halide resin compositions. Fairweather, H. G. C. (General Aniline & Film Corp.). 806 936  
Monoazo dyestuffs of the benzene-azo-naphthalene series. Farbenfabriken Bayer AG. 806 938  
Alkylated bis-phenols and hydroxy benzylamines and their method of manufacture. Ethyl Corp. 806 961  
Production of ethylene copolymers. Farbenfabriken Bayer AG. 807 112  
Polymerisation of ethylene. Hercules Powder Co. 806 904  
Copolymer-oil modified synthetic rubber. Esso Research & Engineering Co. 806 905  
Vapour phase catalytic fluidised reactions. Du Pont de Nemours & Co., E. I. 807 113  
Nicotinic acid esters and process of making same. Lannacher Heilmittel G.m.b.H. 807 115  
Metallic chlorides. Horizons Titanium Corp. 807 116  
Herbicide agents. Du Pont de Nemours & Co., E. I. [Addition to 692 589.] 806 989  
Copolymers of vinyl acetate and fumarate. Esso Research & Engineering Co. 806 906  
Phosphate esters. Bataafsche Petroleum Maatschappij N.V., De. 807 119  
Production of polymerisation products from olefinically-unsaturated hydrocarbons. Badische Anilin- & Soda-Fabrik AG. 807 178  
Pesticides and herbicides. Matieres Colorantes et Produits Chimiques de St. Denis S.A., Des. 807 120

Open to public inspection 14 January 1959

Preparation of chlorides of uranium. Evers, E. C., and Carignan, C. J. 807 261  
Manufacture of carbon and of products derived therefrom. Shell-Refining & Marketing Co., Ltd., Dijk, W. J. D. Van, and Nixon, I. G. 807 221  
Polytrifluorochloroethylene plastic. Miller, W. T. 807 431  
Adhesive compositions. Starch Products, Ltd., and Phillips, E. W. T. 807 505  
Gas-fired water heater. North Thames Gas Board. 807 371, 807 372  
Steroid compounds and processes for the manufacture thereof. Schering Corp. 807 227  
Pyridazine carboxylic acid esters and process of making same. Ciba Ltd. 807 228  
Amine alkylation. Ethyl Corp. 807 229  
Surface-modified silica products. Monsanto Chemical Co. 807 452  
Coating compositions and production thereof. Montanto Chemical Co. [Addition to 807 452 and 807 454.] 807 453  
Halogen-pyridazines and process for their manufacture. Ciba Ltd. 807 548  
Method of improving a refractory article. Armour Research Foundation of Illinois Institute of Technology. [Addition to 776 443.] 807 302  
Method of growing quartz single crystals. Clevite Corp. 807 304  
Chemical compositions useful as blowing agents. Whiffen & Sons Ltd. 807 405  
Extraction vessels. Maschinenfabrik Buckau R. Wolf AG. [Addition to 776 445.] 807 306

Temperature measuring devices. Anderson, J. C. 807 232  
Method of producing olefinic compounds. Institut Francais du Pétrole, Des Carburants et Lubrifiants. 807 501  
Process for preparing gasoline reforming catalysts. Grace & Co., W. R. 807 272  
Pigmented dextran. Commonwealth Engineering Co. of Ohio. 807 234  
Connection for ducts, pipes, conduits and the like. Power Jets (Research & Development), Ltd. 807 336  
Fluorine-containing silanes and siloxanes. Midland Silicones, Ltd. [Addition to 746 510.] 807 235  
Heat-exchange apparatus and processes for the heat treatment of gases. Power Jets (Research & Development), Ltd. 807 418  
Bromine-containing nitrile methylol-phosphorus polymers. Albright & Wilson, Ltd. 807 236  
Automatic filling devices for liquid or plastic substances. Unilever, Ltd. 807 338  
Clarification plant for service and waste waters. Sambras, A. 807 308  
Insulated coatings for magnetic sheet material. General Electric Co. 807 577  
Thermoplastic materials. Styrene Products, Ltd. 807 237  
Protective coatings for articles and a process of preparing said coatings. Knapsack-Griesheim, AG. [Addition to 759 214.] 807 218  
Fluid-pressure gauges. Harcourt, Ltd., D. 807 529  
Process for the preparation of dyestuffs. Farbenfabriken Bayer AG. 807 224  
Steroid compounds. British Drug Houses, Ltd. 807 225  
Collapsible tanks. Unthank, D. G., and Silley, H. A. J. 807 280  
Tubulous vapour generating and vapour heating units. Babcock & Wilcox, Ltd. 807 282  
Polyurethane resins. Pittsburgh Plate Glass Co. 807 377

## DIARY DATES

### MONDAY, 8 DECEMBER

S.C.I., & O.C.C.A.—Hull: Royal Station Hotel, 7 p.m. 'Synthesis of natural fatty acids', by F. D. Gunstone.  
S.C.I.—London: 14 Belgrave Sq., S.W.1. 'Surface activity of organosilicon compounds', by R. L. Bass.

### TUESDAY, 9 DECEMBER

C.S.—Nottingham: Chemistry Dept., University, 5 p.m. 'Chemistry of the tannins', by Prof. R. D. Haworth.  
C.S.—Dundee: Chemistry Dept., Queen's College, 5.5 p.m. 'Effects of size and shape of drug molecules on drug action', by H. R. Ing.  
S.A.C.—London: Feathers, Tudor St., E.C.4., 6.30 p.m. Discussion meeting on 'Methodological reminiscences', opened by S. K. Kon.  
S.A.C.—Nottingham: Gas Showrooms, 7 p.m. 'Analysis of Tar Acids', by H. G. Willcock.  
S.C.I.—London: 14 Belgrave Sq., S.W.1. 6 p.m. 'Patenting of chemical inventions', by J. M. Soesan.  
S.C.I.—London: 14 Belgrave Sq., S.W.1. 10.30 a.m. Forestry meeting.

### WEDNESDAY, 10 DECEMBER

C.S. & R.I.C.—Rotherham: Technical College, 7.30 p.m. 'Some aspects of power station chemistry', by L. A. Huntingdon.  
I.Chem.E.—Birmingham: College of Technology, 6.30 p.m. 'Chemical engineering as applied to manufacture of antibiotics', by J. Stuart.  
S.A.C.—London: Feathers, Tudor St., E.C.4., 6.30 p.m. A discussion meeting, 'Use of microscope in analysis', opened by R. L. Casar.

### THURSDAY, 11 DECEMBER

C.S.—London: Burlington House, W.1., 7.30 p.m. 'Triplet state in chemistry', Tilden lecture by Prof. G. Porter.

### FRIDAY, 12 DECEMBER

R.I.C.—London: Northampton College, St. John St., E.C.1., 6.30 p.m. 'Application of sequestering agents', by J. K. Aiken.



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